

DEMICHEV, A.D.; YINGOVATOV, A.A.; KUZNETSOV, N.N.; KOSTYUKOVICH, N.I.;  
ULYUYEV, D.I.; USHAKOV, S.M.; LIDERS, G.V., kandidat tekhnicheskikh nauk, redaktor; BOEROVA, Ye.N., tekhnicheskiy redaktor

[Mechanizing work in major repairing of railroad tracks; experience of track machinery stations] Mekhanizatsiya rabot po kapital'nomu remontu puti; opyt putevykh mashinnykhstantsii. Moskva, Gos. transp.zhel-dor.izd-vo, 1957. 107 p. (MLRA 10:9)  
(Railroads--Track)

USHAKOV, S.M., inzhener.  
MALITSKIY, L.Ya., inzhener.

The "Matiza" ballast cleaning machine. Put' i put. Knox. no.2:46 P '57.  
(Switzerland--Ballast) (MIRA 10:4)

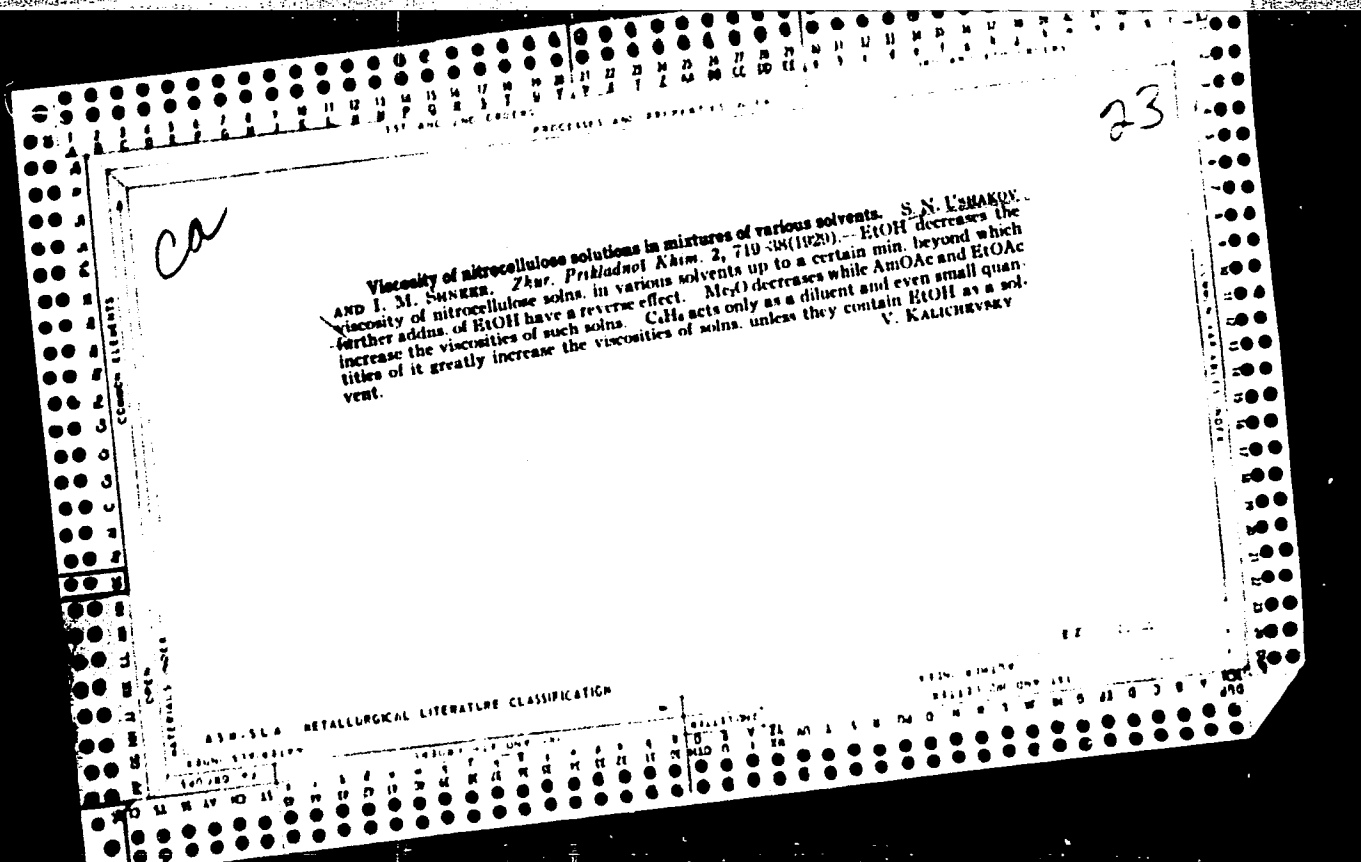
USHAKOV, S.M., inzh.

How to repair the hoisting unit of the B-5 ballast distributor.  
Put' 1 put. khoz. no.3:35-36 Mr '58. (MIRA 11:4)  
(Ballast (Railroads))

USHAKOV, S.M.; "zasluzhennyi deyatel' nauki i tekhniki RSFSR, Leningrad.

Polymers in medicine. Nauka i zhytiia 11 no.2:28 F '61.  
(MIRA 14:3)

1. Chlen-korrespondent AN SSSR.  
(POLYMERS) (MEDICAL SUPPLIES)



22

CA

1ST AND 2ND ORDERS PROCESSES AND PROPERTIES INDEX

The synthesis of mixed benzyl ethyl ethers of cellulose. S. N. Ushakov and N. N. Krestinskaya. *Plasticheskie Massy* 1934, No. 1, 8-13. Cellulose is allowed to stand with 1 mol. excess of 50% NaOH. Part of the H<sub>2</sub>O is distilled from the mixt., which is then heated in an autoclave with 6-10 mols. of EtCl and 0.2-0.3 mol. of PhCH<sub>2</sub>Cl in C<sub>6</sub>H<sub>6</sub> soln. for 16 hrs. at 130°. Increase in the amt. of NaOH used increases the soly. of the product in C<sub>6</sub>H<sub>6</sub>, but weakens it mechanically. Increase in the amt. of PhCH<sub>2</sub>Cl increases the stability of the product to H<sub>2</sub>O, but lowers the viscosity and mech. strength. The mixed ether is more resistant to H<sub>2</sub>O than pure ethylcellulose. Benzylolation of partially ethylated cellulose gives an unsatisfactory product. H. M. Leikreter

COMMON ELEMENTS

ALPHABETIC INDEX

ASAC-36.4 METALLURGICAL LITERATURE CLASSIFICATION

FROM SYNOPSIS

197005 1

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46 47 48 49 50 51 52 53 54 55 56 57 58 59 60 61 62 63 64 65 66 67 68 69 70 71 72 73 74 75 76 77 78 79 80 81 82 83 84 85 86 87 88 89 90 91 92 93 94 95 96 97 98 99 100

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46 47 48 49 50 51 52 53 54 55 56 57 58 59 60 61 62 63 64 65 66 67 68 69 70 71 72 73 74 75 76 77 78 79 80 81 82 83 84 85 86 87 88 89 90 91 92 93 94 95 96 97 98 99 100

23

The synthesis of propylcellulose. S. N. Ushakov and S. I. Kucherenko. *Plasticheskie Massy* 1994, No. 3, 12-17. A 1:10 mixt. of mercerized cellulose and  $\text{PrCl}$  heated 12 hrs. at  $140-160^\circ$  gives a propylcellulose with 1.5 1.75 OH groups replaced by Pr. Increase in reaction time to 24 hrs. gives a product of poorer quality. Increase in the ratio of reactants to 1:20 gives a substitution of OH groups up to 2.25. The more highly substituted ethers are less hygroscopic and stronger than the less substituted ones. The mech. and chem. properties in general are typical of those of cellulose ethers. H. M. Leicester

ASB-SLA METALLURGICAL LITERATURE CLASSIFICATION

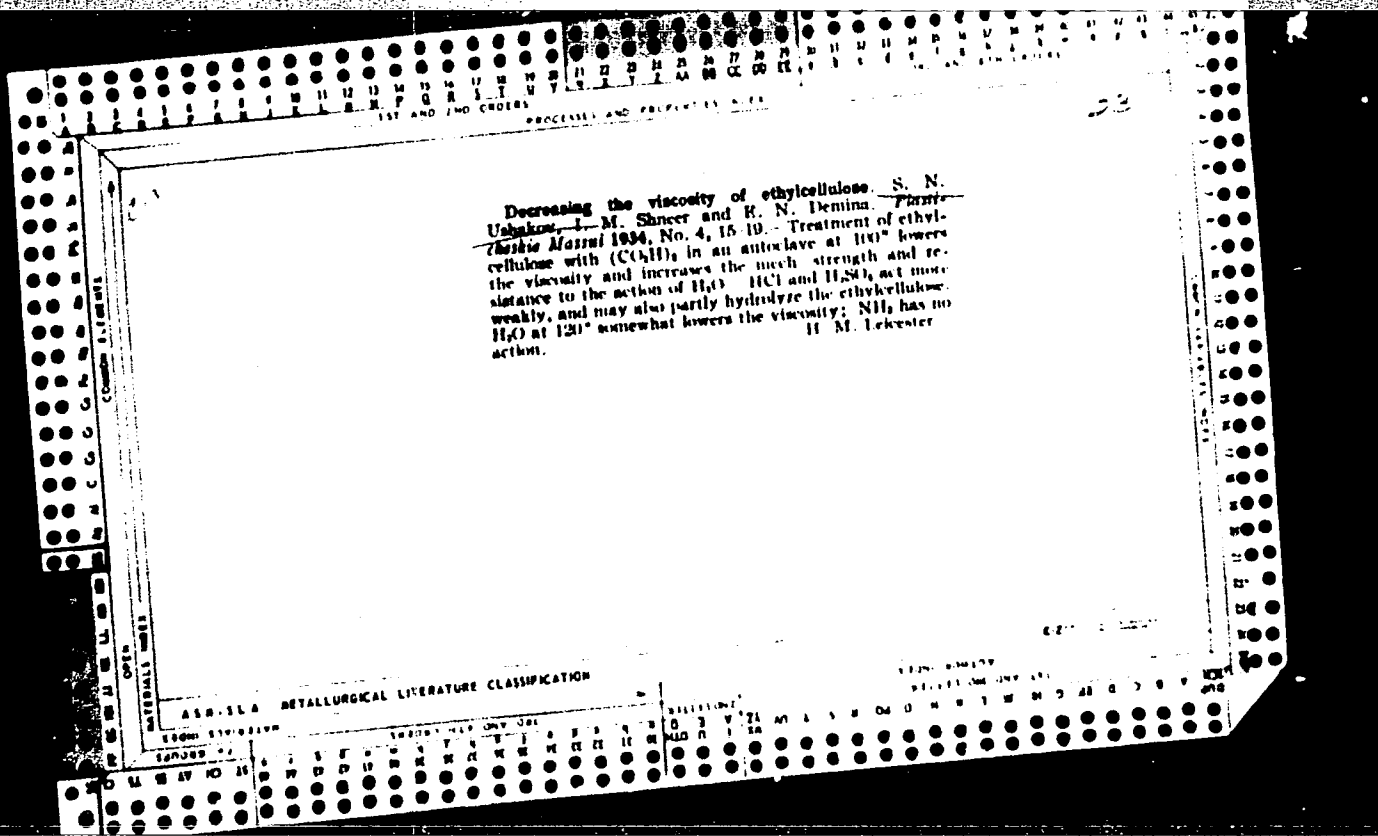
REGION 1

REGION 2

REGION 3

REGION 4

REGION 5





B-II-6

**Synthesis of butyrolactone:** S. N. Ushakov and  
Y. A. Kozlova (Plast. Mass, 1964, No. 6, 1-3). — BuCl  
(10 mole), succinyl chloride (1 mole), and NaOH (3  
mole) in 40-50% solution are heated together for  
12 hr. at 120-130°. The butyrolactone formed (yield  
80-85% of theory) contains 8-9.5 Ba groups. Reason  
of failure to obtain the preparation of the product. Ch. Acc. (p)

**AIN-11A METALLURGICAL LITERATURE CLASSIFICATION**

FROM SYMBOL	TO SYMBOL	RELATIONS	FROM SYMBOL	TO SYMBOL
10	11	12	13	14
15	16	17	18	19
20	21	22	23	24
25	26	27	28	29
30	31	32	33	34
35	36	37	38	39
40	41	42	43	44
45	46	47	48	49
50	51	52	53	54
55	56	57	58	59
60	61	62	63	64
65	66	67	68	69
70	71	72	73	74
75	76	77	78	79
80	81	82	83	84
85	86	87	88	89
90	91	92	93	94
95	96	97	98	99
100	101	102	103	104
105	106	107	108	109
110	111	112	113	114
115	116	117	118	119
120	121	122	123	124
125	126	127	128	129
130	131	132	133	134
135	136	137	138	139
140	141	142	143	144
145	146	147	148	149
150	151	152	153	154
155	156	157	158	159
160	161	162	163	164
165	166	167	168	169
170	171	172	173	174
175	176	177	178	179
180	181	182	183	184
185	186	187	188	189
190	191	192	193	194
195	196	197	198	199
200	201	202	203	204
205	206	207	208	209
210	211	212	213	214
215	216	217	218	219
220	221	222	223	224
225	226	227	228	229
230	231	232	233	234
235	236	237	238	239
240	241	242	243	244
245	246	247	248	249
250	251	252	253	254
255	256	257	258	259
260	261	262	263	264
265	266	267	268	269
270	271	272	273	274
275	276	277	278	279
280	281	282	283	284
285	286	287	288	289
290	291	292	293	294
295	296	297	298	299
300	301	302	303	304
305	306	307	308	309
310	311	312	313	314
315	316	317	318	319
320	321	322	323	324
325	326	327	328	329
330	331	332	333	334
335	336	337	338	339
340	341	342	343	344
345	346	347	348	349
350	351	352	353	354
355	356	357	358	359
360	361	362	363	364
365	366	367	368	369
370	371	372	373	374
375	376	377	378	379
380	381	382	383	384
385	386	387	388	389
390	391	392	393	394
395	396	397	398	399
400	401	402	403	404
405	406	407	408	409
410	411	412	413	414
415	416	417	418	419
420	421	422	423	424
425	426	427	428	429
430	431	432	433	434
435	436	437	438	439
440	441	442	443	444
445	446	447	448	449
450	451	452	453	454
455	456	457	458	459
460	461	462	463	464
465	466	467	468	469
470	471	472	473	474
475	476	477	478	479
480	481	482	483	484
485	486	487	488	489
490	491	492	493	494
495	496	497	498	499
500	501	502	503	504
505	506	507	508	509
510	511	512	513	514
515	516	517	518	519
520	521	522	523	524
525	526	527	528	529
530	531	532	533	534
535	536	537	538	539
540	541	542	543	544
545	546	547	548	549
550	551	552	553	554
555	556	557	558	559
560	561	562	563	564
565	566	567	568	569
570	571	572	573	574
575	576	577	578	579
580	581	582	583	584
585	586	587	588	589
590	591	592	593	594
595	596	597	598	599
600	601	602	603	604
605	606	607	608	609
610	611	612	613	614
615	616	617	618	619
620	621	622	623	624
625	626	627	628	629
630	631	632	633	634
635	636	637	638	639
640	641	642	643	644
645	646	647	648	649
650	651	652	653	654
655	656	657	658	659
660	661	662	663	664
665	666	667	668	669
670	671	672	673	674
675	676	677	678	679
680	681	682	683	684
685	686	687	688	689
690	691	692	693	694
695	696	697	698	699
700	701	702	703	704
705	706	707	708	709
710	711	712	713	714
715	716	717	718	719
720	721	722	723	724
725	726	727	728	729
730	731	732	733	734
735	736	737	738	739
740	741	742	743	744
745	746	747	748	749
750	751	752	753	754
755	756	757	758	759
760	761	762	763	764
765	766	767	768	769
770	771	772	773	774
775	776	777	778	779
780	781	782	783	784
785	786	787	788	789
790	791	792	793	794
795	796	797	798	799
800	801	802	803	804
805	806	807	808	809
810	811	812	813	814
815	816	817	818	819
820	821	822	823	824
825	826	827	828	829
830	831	832	833	834
835	836	837	838	839
840	841	842	843	844
845	846	847	848	849
850	851	852	853	854
855	856	857	858	859
860	861	862	863	864
865	866	867	868	869
870	871	872	873	874
875	876	877	878	879
880	881	882	883	884
885	886	887	888	889
890	891	892	893	894
895	896	897	898	899
900	901	902	903	904
905	906	907	908	909
910	911	912	913	914
915	916	917	918	919
920	921	922	923	924
925	926	927	928	929
930	931	932	933	934
935	936	937	938	939
940	941	942	943	944
945	946	947	948	949
950	951	952	953	954
955	956	957	958	959
960	961	962	963	964
965	966	967	968	969
970	971	972	973	974
975	976	977	978	979
980	981	982	983	984
985	986	987	988	989
990	991	992	993	994
995	996	997	998	999
1000	1001	1002	1003	1004
1005	1006	1007	1008	1009
1010	1011	1012	1013	1014
1015	1016	1017	1018	1019
1020	1021	1022	1023	1024
1025	1026	1027	1028	1029
1030	1031	1032	1033	1034
1035	1036	1037	1038	1039
1040	1041	1042	1043	1044
1045	1046	1047	1048	1049
1050	1051	1052	1053	1054
1055	1056	1057	1058	1059
1060	1061	1062	1063	1064
1065	1066	1067	1068	1069
1070	1071	1072	1073	1074
1075	1076	1077	1078	1079
1080	1081	1082	1083	1084
1085	1086	1087	1088	1089
1090	1091	1092	1093	1094
1095	1096	1097	1098	1099
1100	1101	1102	1103	1104
1105	1106	1107	1108	1109
1110	1111	1112	1113	1114
1115	1116	1117	1118	1119
1120	1121	1122	1123	1124
1125	1126	1127	1128	1129
1130	1131	1132	1133	1134
1135	1136	1137	1138	1139
1140	1141	1142	1143	1144
1145	1146	1147	1148	1149
1150	1151	1152	1153	1154

1st and 2nd covers

SYNTHESIS AND PREPARATION

68

Synthetic resins in the lacquer industry and different methods for their preparation in the U. S. R. S. N. Ushakov. *Lakobrasochnyy Industriyn* 1934, No. 5 0. 35-6. H. M. Leicester

COMMON ELEMENTS

12345678910111213141516171819202122232425262728293031323334353637383940414243444546474849505152535455565758596061626364656667686970717273747576777879808182838485868788899091929394959697989900

ASB-SLA METALLURGICAL LITERATURE CLASSIFICATION

12345678910111213141516171819202122232425262728293031323334353637383940414243444546474849505152535455565758596061626364656667686970717273747576777879808182838485868788899091929394959697989900

TEST AND PROPERTIES INDEX																									
TEST AND PROPERTIES INDEX													TEST AND PROPERTIES INDEX												
<p>Possibilities for the use of sapropel in plastic compounds. S. N. Ushakov, G. S. Brodskii and E. G. Ososvskaya. <i>Plasticheskie Massy</i> 1934, No. 6, 28-9.</p> <p>Plastics prepd. from sapropel have rather poor properties. H. M. Leicester</p>																									
<p>AS 4-51 A METALLURGICAL LITERATURE CLASSIFICATION</p>																									
<p>REGION: 51-51-111</p>																									
<p>CLASSIFICATION: 51-51-111</p>																									
<p>CLASSIFICATION: 51-51-111</p>																									

**Compositions from polymeric vinyl compounds and phenol-aldehyde resins.** S. N. Lashkov, Russ. 42,1590, April 30, 1935. The compounds are prepared by adding a diluent to the solns., such as heavy gasoline, which does not dissolve the polymeric vinyl compd. and phenol-aldehyde resins, but which dissolves the phenol-aldehyde resin in a mixt. with the original vinyl compd.

ASB.SLA METALLURGICAL LITERATURE CLASSIFICATION

10

Salts of benzylideneacetic acid. M. N. Likhachev and  
A. A. Rozenfeld. Russ. 44,547, TRF. 31, 1935. A  
soln. of salts of benzylideneacetic acid is heated with benzyl  
chloride. The product is useful as a plasticizing agent.



1ST AND 2ND ORDERS																										3RD AND 4TH ORDERS																									
MATERIALS INDEX																										COMMON VARIABLE INDEX																									
CA																										13																									
<p>The synthesis of oil-soluble artificial phenol resins of the type of albertol. S. N. Ushakov and V. M. Zolotarev. <i>Narodnyi Komissariat Tishkol Prom. S. S. S. R. Leningrad, Plastmassy</i> 1, 233-40 (1930). Albertol can be prepd. from 60 parts of abietic acid heated together for 5 hrs. and 50 parts of glycerol heated together for 5 hrs., and then esterified with 8.5 parts of 24% H<sub>2</sub>O. glycerol for 7-8 hrs. at 250-60°. These amts. can be varied in wide limits without greatly affecting the product, but if more than 125 parts of CH<sub>2</sub>O soln. is used, the product is insol. in oils. Albertol can also be prepd. by heating 100 parts of iditol with 200 parts of natural resin at 200° for 24-6 hrs. Addn. of EtOH to C<sub>6</sub>H<sub>6</sub> or Et<sub>2</sub>O solns. of albertol raises the acid no. H. M. Leicester</p>																																																			
<p>ASM-SLA METALLURGICAL LITERATURE CLASSIFICATION</p>																																																			
<p>GROUPS: 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46 47 48 49 50 51 52</p>																																																			

13

CA

The conditions for the condensation of different portions of shale tars with formaldehyde to form products similar to resol. S. N. Ushakov and G. S. Brudskii. *Narodnii Komissariat Tysakhiol Prom. S. S. R., Leningrad, Plastmassul 1, 233, 300 (1935).*—The PhOH portion from shale oil, b 170-320°, gives satisfactory resins with  $\text{CH}_2\text{O}$  when  $\text{NaOH}$  or  $\text{K}_2\text{CO}_3$  is used as catalyst. Residual high-boiling neutral oil in the resin acts as a plasticizer, but tends to decompose at high temp. and pressure.

H. M. Leicester



**The condensation of acetaldehyde.** S. N. Ushakov and I. G. Shitan'ko. *Narodnui Komissariat Tyuzhelei Prom*. S. S. S. R., Leningrad, *Plastmassy* 1, 860-42 (1935). — AcHl is best polymerized to a soft tar by stirring it with a 10-20% NaOH soln. contg. 1.5% of the wt. of AcHl of NaOH for 10-12 hrs. Higher NaOH concns. increase the reaction rate, but give darker products. When this tar is heated at 180-200°, the m. p. rises and the soly. decreases, so that by stopping the heating at different periods, resins with different properties can be obtained. Heating in a stream of O darkens the product. Addn. of H<sub>2</sub>O acids before condensation gives a more elastic product, but the m. p. is lower and the soly. is less. Addn. of metallic oxides hardens the resins, but lowers the soly. in org. solvents. Chlorination of the tar gives a very poor product. Addn. of (MeC<sub>2</sub>H<sub>5</sub>)<sub>3</sub>PO, permits formation of good films. H. M. Leicester

USHAKOV, S. N.

The phenol-lignin resins. S. N. Ushakov, L. I. Matveev, and O. E. Iv. Lesokhin. From. 1939, No. 1, 23-31; Khim. Referat, Zhur, 1939, No. 8, 111.--In condensation of phenol with tech. lignin (freed from cellulose and pentosans) by treatment for 14-15 hrs. at 135-140° and then for 4 hrs. at 170-180° with 5% of H<sub>2</sub>SO<sub>4</sub> on the wt. of phenol, approx. 1 mol. of water is sepd. for each mol. of phenol. In general this verifies the reaction mechanism proposed by Wedekind. At 115-120°, up to 14% of lignin (on the wt. of phenol) can be added to the reaction mixt. Addn. of lignin to highly heated phenol causes much foam formation. At a lower temp. the amt. of lignin which can be added is considerably less. The optimum amt. of the catalyst (H<sub>2</sub>SO<sub>4</sub>) is 2.5% and the optimum time of condensation (phenol:lignin = 100:100) is 3 hrs. With the ratio 100:140 the optimum time is 5 hrs. The moisture content of the lignin should not exceed 15%. Approx. 2 parts by wt. of lignin is bound by 1 part of phenol. The m. p. of the resins (depending on their phenol contents) varies from 40 to 130°. They are sol. in alc. to the extent of 78-88%, in acetone to 90-2%; they are insol. in benzene and ether. They are similar to novolak resins.

W. R. Henn

USHAKOV, S. N.

Resins and plastic masses. S. N. Ushakov, U.S.S.R.  
64,571, April 30, 1946. A fusion of phenol and alkali  
lignin is mixed with hexamethylenetetramine or with a  
solid polymer of  $\text{CH}_2\text{O}$ . In order to obtain an infusible  
and insol. end product, the quantity of hexamethylene-  
tetramine is approx. 10% of the fusion mass. M. Hosen

22

CA

PROCESSES AND PROPERTIES INDEX

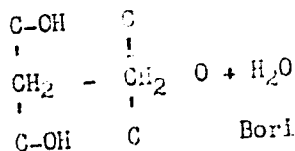
The acid fraction of generator tar from Baltic shales. S. N. Ushakov, B. I. Ivanov, and S. I. Kirillova. *Khim. Prom.* 1946, No. 9, 11-15. This study concerned the suitability of the acid fraction of tar obtained in the gasification of Baltic shales for the manuf. of plastics. From the tar 2 fractions were sepd.: one b. up to 230° and the other b. 230-325°. The first of these amounted to 15% and contained 4.9% phenols. The second amounted to 31% and contained 23% phenols. Phenols sepd. from the phenolates of the benzene fraction of the tar were also studied. The phenols consisted mainly of cresols, to a lesser extent xylenols, and an insignificant quantity of carbolic acid. The phenols were tried in various combinations with  $\text{CH}_3\text{O}$  for producing synthetic resins with acid and alk. catalysts. M. Hosh

ASB-SLA METALLURGICAL LITERATURE CLASSIFICATION

33000 33100 33200 33300 33400 33500 33600 33700 33800 33900 34000 34100 34200 34300 34400 34500 34600 34700 34800 34900 35000 35100 35200 35300 35400 35500 35600 35700 35800 35900 36000 36100 36200 36300 36400 36500 36600 36700 36800 36900 37000 37100 37200 37300 37400 37500 37600 37700 37800 37900 38000 38100 38200 38300 38400 38500 38600 38700 38800 38900 39000 39100 39200 39300 39400 39500 39600 39700 39800 39900 40000 40100 40200 40300 40400 40500 40600 40700 40800 40900 41000 41100 41200 41300 41400 41500 41600 41700 41800 41900 42000 42100 42200 42300 42400 42500 42600 42700 42800 42900 43000 43100 43200 43300 43400 43500 43600 43700 43800 43900 44000 44100 44200 44300 44400 44500 44600 44700 44800 44900 45000 45100 45200 45300 45400 45500 45600 45700 45800 45900 46000 46100 46200 46300 46400 46500 46600 46700 46800 46900 47000 47100 47200 47300 47400 47500 47600 47700 47800 47900 48000 48100 48200 48300 48400 48500 48600 48700 48800 48900 49000 49100 49200 49300 49400 49500 49600 49700 49800 49900 50000 50100 50200 50300 50400 50500 50600 50700 50800 50900 51000 51100 51200 51300 51400 51500 51600 51700 51800 51900 52000 52100 52200 52300 52400 52500 52600 52700 52800 52900 53000 53100 53200 53300 53400 53500 53600 53700 53800 53900 54000 54100 54200 54300 54400 54500 54600 54700 54800 54900 55000 55100 55200 55300 55400 55500 55600 55700 55800 55900 56000 56100 56200 56300 56400 56500 56600 56700 56800 56900 57000 57100 57200 57300 57400 57500 57600 57700 57800 57900 58000 58100 58200 58300 58400 58500 58600 58700 58800 58900 59000 59100 59200 59300 59400 59500 59600 59700 59800 59900 60000 60100 60200 60300 60400 60500 60600 60700 60800 60900 61000 61100 61200 61300 61400 61500 61600 61700 61800 61900 62000 62100 62200 62300 62400 62500 62600 62700 62800 62900 63000 63100 63200 63300 63400 63500 63600 63700 63800 63900 64000 64100 64200 64300 64400 64500 64600 64700 64800 64900 65000 65100 65200 65300 65400 65500 65600 65700 65800 65900 66000 66100 66200 66300 66400 66500 66600 66700 66800 66900 67000 67100 67200 67300 67400 67500 67600 67700 67800 67900 68000 68100 68200 68300 68400 68500 68600 68700 68800 68900 69000 69100 69200 69300 69400 69500 69600 69700 69800 69900 70000 70100 70200 70300 70400 70500 70600 70700 70800 70900 71000 71100 71200 71300 71400 71500 71600 71700 71800 71900 72000 72100 72200 72300 72400 72500 72600 72700 72800 72900 73000 73100 73200 73300 73400 73500 73600 73700 73800 73900 74000 74100 74200 74300 74400 74500 74600 74700 74800 74900 75000 75100 75200 75300 75400 75500 75600 75700 75800 75900 76000 76100 76200 76300 76400 76500 76600 76700 76800 76900 77000 77100 77200 77300 77400 77500 77600 77700 77800 77900 78000 78100 78200 78300 78400 78500 78600 78700 78800 78900 79000 79100 79200 79300 79400 79500 79600 79700 79800 79900 80000 80100 80200 80300 80400 80500 80600 80700 80800 80900 81000 81100 81200 81300 81400 81500 81600 81700 81800 81900 82000 82100 82200 82300 82400 82500 82600 82700 82800 82900 83000 83100 83200 83300 83400 83500 83600 83700 83800 83900 84000 84100 84200 84300 84400 84500 84600 84700 84800 84900 85000 85100 85200 85300 85400 85500 85600 85700 85800 85900 86000 86100 86200 86300 86400 86500 86600 86700 86800 86900 87000 87100 87200 87300 87400 87500 87600 87700 87800 87900 88000 88100 88200 88300 88400 88500 88600 88700 88800 88900 89000 89100 89200 89300 89400 89500 89600 89700 89800 89900 90000 90100 90200 90300 90400 90500 90600 90700 90800 90900 91000 91100 91200 91300 91400 91500 91600 91700 91800 91900 92000 92100 92200 92300 92400 92500 92600 92700 92800 92900 93000 93100 93200 93300 93400 93500 93600 93700 93800 93900 94000 94100 94200 94300 94400 94500 94600 94700 94800 94900 95000 95100 95200 95300 95400 95500 95600 95700 95800 95900 96000 96100 96200 96300 96400 96500 96600 96700 96800 96900 97000 97100 97200 97300 97400 97500 97600 97700 97800 97900 98000 98100 98200 98300 98400 98500 98600 98700 98800 98900 99000 99100 99200 99300 99400 99500 99600 99700 99800 99900 100000 100100 100200 100300 100400 100500 100600 100700 100800 100900 101000 101100 101200 101300 101400 101500 101600 101700 101800 101900 102000 102100 102200 102300 102400 102500 102600 102700 102800 102900 103000 103100 103200 103300 103400 103500 103600 103700 103800 103900 104000 104100 104200 104300 104400 104500 104600 104700 104800 104900 105000 105100 105200 105300 105400 105500 105600 105700 105800 105900 106000 106100 106200 106300 106400 106500 106600 106700 106800 106900 107000 107100 107200 107300 107400 107500 107600 107700 107800 107900 108000 108100 108200 108300 108400 108500 108600 108700 108800 108900 109000 109100 109200 109300 109400 109500 109600 109700 109800 109900 110000 110100 110200 110300 110400 110500 110600 110700 110800 110900 111000 111100 111200 111300 111400 111500 111600 111700 111800 111900 112000 112100 112200 112300 112400 112500 112600 112700 112800 112900 113000 113100 113200 113300 113400 113500 113600 113700 113800 113900 114000 114100 114200 114300 114400 114500 114600 114700 114800 114900 115000 115100 115200 115300 115400 115500 115600 115700 115800 115900 116000 116100 116200 116300 116400 116500 116600 116700 116800 116900 117000 117100 117200 117300 117400 117500 117600 117700 117800 117900 118000 118100 118200 118300 118400 118500 118600 118700 118800 118900 119000 119100 119200 119300 119400 119500 119600 119700 119800 119900 120000 120100 120200 120300 120400 120500 120600 120700 120800 120900 121000 121100 121200 121300 121400 121500 121600 121700 121800 121900 122000 122100 122200 122300 122400 122500 122600 122700 122800 122900 123000 123100 123200 123300 123400 123500 123600 123700 123800 123900 124000 124100 124200 124300 124400 124500 124600 124700 124800 124900 125000 125100 125200 125300 125400 125500 125600 125700 125800 125900 126000 126100 126200 126300 126400 126500 126600 126700 126800 126900 127000 127100 127200 127300 127400 127500 127600 127700 127800 127900 128000 128100 128200 128300 128400 128500 128600 128700 128800 128900 129000 129100 129200 129300 129400 129500 129600 129700 129800 129900 130000 130100 130200 130300 130400 130500 130600 130700 130800 130900 131000 131100 131200 131300 131400 131500 131600 131700 131800 131900 132000 132100 132200 132300 132400 132500 132600 132700 132800 132900 133000 133100 133200 133300 133400 133500 133600 133700 133800 133900 134000 134100 134200 134300 134400 134500 134600 134700 134800 134900 135000 135100 135200 135300 135400 135500 135600 135700 135800 135900 136000 136100 136200 136300 136400 136500 136600 136700 136800 136900 137000 137100 137200 137300 137400 137500 137600 137700 137800 137900 138000 138100 138200 138300 138400 138500 138600 138700 138800 138900 139000 139100 139200 139300 139400 139500 139600 139700 139800 139900 140000 140100 140200 140300 140400 140500 140600 140700 140800 140900 141000 141100 141200 141300 141400 141500 141600 141700 141800 141900 142000 142100 142200 142300 142400 142500 142600 142700 142800 142900 143000 143100 143200 143300 143400 143500 143600 143700 143800 143900 144000 144100 144200 144300 144400 144500 144600 144700 144800 144900 145000 145100 145200 145300 145400 145500 145600 145700 145800 145900 146000 146100 146200 146300 146400 146500 146600 146700 146800 146900 147000 147100 147200 147300 147400 147500 147600 147700 147800 147900 148000 148100 148200 148300 148400 148500 148600 148700 148800 148900 149000 149100 149200 149300 149400 149500 149600 149700 149800 149900 150000 150100 150200 150300 150400 150500 150600 150700 150800 150900 151000 151100 151200 151300 151400 151500 151600 151700 151800 151900 152000 152100 152200 152300 152400 152500 152600 152700 152800 152900 153000 153100 153200 153300 153400 153500 153600 153700 153800 153900 154000 154100 154200 154300 154400 154500 154600 154700 154800 154900 155000 155100 155200 155300 155400 155500 155600 155700 155800 155900 156000 156100 156200 156300 156400 156500 156600 156700 156800 156900 157000 157100 157200 157300 157400 157500 157600 157700 157800 157900 158000 158100 158200 158300 158400 158500 158600 158700 158800 158900 159000 159100 159200 159300 159400 159500 159600 159700 159800 159900 160000 160100 160200 160300 160400 160500 160600 160700 160800 160900 161000 161100 161200 161300 161400 161500 161600 161700 161800 161900 162000 162100 162200 162300 162400 162500 162600 162700 162800 162900 163000 163100 163200 163300 163400 163500 163600 163700 163800 163900 164000 164100 164200 164300 164400 164500 164600 164700 164800 164900 165000 165100 165200 165300 165400 165500 165600 165700 165800 165900 166000 166100 166200 166300 166400 166500 166600 166700 166800 166900 167000 167100 167200 167300 167400 167500 167600 167700 167800 167900 168000 168100 168200 168300 168400 168500 168600 168700 168800 168900 169000 169100 169200 169300 169400 169500 169600 169700 169800 169900 170000 170100 170200 170300 170400 170500 170600 170700 170800 170900 171000 171100 171200 171300 171400 171500 171600 171700 171800 171900 172000 172100 172200 172300 172400 172500 172600 172700 172800 172900 173000 173100 173200 173300 173400 173500 173600 173700 173800 173900 174000 174100 174200 174300 174400 174500 174600 174700 174800 174900 175000 175100 175200 175300 175400 175500 175600 175700 175800 175900 176000 176100 176200 176300 176400 176500 176600 176700 176800 176900 177000 177100 177200 177300 177400 177500 177600 177700 177800 177900 178000 178100 178200 178300 178400 178500 178600 178700 178800 178900 179000 179100 179200 179300 179400 179500 179600 179700 179800 179900 180000 180100 180200 180300 180400 180500 180600 180700 180800 180900 181000 181100 181200 181300 181400 181500 181600 181700 181800 181900 182000 182100 182200 182300 182400 182500 182600 182700 182800 182900 183000 183100 183200 183300 183400 183500 183600 183700 183800 183900 184000 184100 184200 184300 184400 184500 184600 184700 184800 184900 185000 185100 185200 185300 185400 185500 185600 185700 185800 185900 186000 186100 186200 186300 186400 186500 186600 186700 186800 186900 187000 187100 187200 187300 187400 187500 187600 187700 187800 187900 188000 188100 188200 188300 188400 188500 188600 188700 188800 188900 189000 189100 189200 189300 189400 189500 189600 189700 189800 189900 190000 190100 190200 190300 190400 190500 190600 190700 190800 190900 191000 191100 191200 191300 191400 191500 191600 191700 191800 191900 192000 192100 192200 192300 192400 192500 192600 192700 192800 192900 193000 193100 193200 193300 193400 193500 193600 193700 193800 193900 194000 194100 194200 194300 194400 194500 194600 194700 194800 194900 195000 195100 195200 195300 195400 195500 195600 195700 195800 195900 196000 196100 196200 196300 196400 196500 196600 196700 196800 196900 197000 197100 197200 197300 197400 197500 197600 197700 197800 197900 198000 198100 198200 198300 198400 198500 198600 198700 198800 198900 199000 199100 199200 199300 199400 199500 199600 199700 199800 199900 200000 200100 200200 200300 200400 200500 200600 200700 200800 200900 201000 201100 201200 201300 201400 201500 201600 201700 201800 201900 202000 202100 202200 202300 202400 202500 202600 202700 202800 202900 203000 203100 203200 203300 203400 203500 203600 203700 203800 203900 204000 204100 204200 204300 204400 204500 204600 204700 204800 204900 205000 205100 205200 205300 205400 205500 205600 205700 205800 205900 206000 206100 206200 206300 206400 206500 206600 206700 206800 206900 207000 207100 207200 207300 207400 207500 207600 207700 207800 207900 208000 208100 208200 208300 208400 208500 208600 208700 208800 208900 209000 209100 209200 209300 209400 209500 209600 209700 209800 209900 210000 210100 210200 210300 210400 210500 210600 210700 210800 210900 211000 211100 211200 211300 211400 211500 211600 211700 211800 211900 212000 212100 212200 212300 212400 212500 212600 212700 212800 212900 213000 2

USHAKOV, S. N.

Synthesis of polyvinylbutyral in a heterogeneous medium. ~~S. N. Ushakov, I. A. Arbuzova,~~  
and E. N. Rostovskii. J. Applied Chem. (U.S.S.R.) 19, 128-33 (1946).—Polyvinyl alc. (I)  
was prepd. by hydrolysis of a 25% alc. soln. of the acetate with 10-13% H<sub>2</sub>SO<sub>4</sub>; polyvinyl  
formate is readily hydrolyzed in aq. medium. The acetalization took place very readily  
in aq. medium by dissolving 1-2 g. I, with 1.04-1.72 g. HCO<sub>2</sub>H as catalyst, and 0.54 ol.  
of PrCHO in 10-27 vols. of water and heating to 40-60° 1-6.5 hrs; 1.27-2.5 g. of poly-  
vinylbutyral, with 74-90% substitution, were obtained. The resultant product was lumpy  
with the lower, and a fine white powder with the higher amt. of water. Lower temps. gave  
a product with a lower acetal content and less aldehyde, swelling in water and filtering  
with difficulty. Adding 1% H<sub>2</sub>SO<sub>4</sub> to 1 g. I in 17-20 ml. water, 1.04 g. HCO<sub>2</sub>H. and  
0.35-0.62 g. PrCHO gave 1.03-1.08 g. of a fine powder (representing 60.9-74% substitution)  
which became sticky on drying. Refluxing 2-5 g. I and a 0-1.2% acid solu. for 1 hrs. in  
benzene yielded 0.1-0.6 g. H<sub>2</sub>O; continuing 12 to 4 hrs. longer in xylene gave and addnl.  
0.42-1.1 g. H<sub>2</sub>O. The authors postulate the formation of an anhydride, as the water  
collected corresponded to the theoretical amt. according to the reaction shown below: the  
presence of acid apparently accelerated it. Under the conditions of the reaction, I is a  
surface-active agent and foams strongly; the reaction takes place on the surface, leading  
to the gradual transformation of the foam to a solid aggregate of the acetal, depending  
on the concn.



Boris Gutoff

**Synthesis of polyvinylformal in a homogeneous medium.**  
 S. N. Lashuk and O. B. Iv (Leningrad Chem. Technol. Inst.). *J. Applied Chem. (U.S.S.R.)* 19, 853-60 (1946) (in Russian).—A "single-tank" method was investigated of hydrolysis of polyvinyl acetate (I) and acetalation of the resulting polyvinyl alc. (II) with HCHO in AcOH soln.; the process was conducted with I of  $\eta$  0.18 poise, 1 mol. soln. in  $\text{C}_2\text{H}_5$ , and a 32% soln. of HCHO, with  $\text{H}_2\text{SO}_4$  (d. 1.84) as catalyst. At 15°, I is only sol. in AcOH of at least 50%. The reaction was started by heating I with concd. AcOH on the water bath for 0.5-1.0 hr. until all was dissolved, after which  $\text{H}_2\text{SO}_4$  and HCHO were added; in one series of expts., water was also added to dil. AcOH from 60 to 60%. Depending on the viscosity of the soln., II is pptd. in grains, fibers, or powder; to obtain a fine ppt., it is necessary to dil. in advance with AcOH (of not less than 50%) until its concn. is 7-8% with respect to I; this dil. soln. is poured into water and II is pptd. in fine threads; pptn. can also be achieved by adding water to the soln. until AcOH is about 30-35%. The yield in II was 95-98%; the product is an incomplete formal, contg. also OH and AcO functions. With 3%  $\text{H}_2\text{SO}_4$ , 20% I in soln., and 1 mole HCHO in 60% AcOH, the degree of substitution  $\alpha$  (moles of substituent per 100 moles of monomer) increased regularly with the time of reaction with regard to the formal group (75.9 and 84.4 after 5 and 25 hrs., resp.), decreased with regard to OH and AcO, reaching equil. after about 25 hrs.; with 82-92% AcOH,  $\alpha$  for AcO falls for the first 5-6 hrs., then increases again; for OH,  $\alpha$  decreases regularly; for the formal group, correspondingly,  $\alpha$  increases during 8 hrs.,

then decreases slightly (16 hrs.). Increase in the amt. of HCHO above the theoretical 0.5 mole per mole monomer shifts the equil. in favor of formal; e.g., 10% AcOH, 15 hrs. HCHO 0.5 and 1.0 moles,  $\alpha$  (formal) 80.2 and 88.2; further increase in HCHO has no further effect. Variation of the amt. of  $\text{H}_2\text{SO}_4$  (1, 3, 5%) is without effect on  $\alpha$ , owing to the fact that while more dil.  $\text{H}_2\text{SO}_4$  favors substitution of OH by formal, its hydrolytic action on I is slower, and vice versa. Variation of the temp. (75 and 100°) has no effect on  $\alpha$  of the finished product even though the higher temp. accelerates somewhat the reaction in its initial stages. Higher concn. of AcOH accelerates the reaction considerably; e.g., for 60, 75, 82, and 90%  $\alpha$  (formal) is 79.3 in 20 hrs., 80.6 in 16 hrs., 84.4 in 6 hrs., 83.0 in 4 hrs. In AcOH stronger than 80%, acetate is being reformed along with formal, the first reaction being catalyzed by  $\text{H}_2\text{SO}_4$ ; under these conditions, a higher  $\alpha$  for formal is reached without  $\text{H}_2\text{SO}_4$  than in its presence (80.1 against 85.8, in 16 hrs., 10% I, 0.75 mole HCHO, mole monomer, 0 and 5%  $\text{H}_2\text{SO}_4$ ). The amt. of I in the soln. can be increased up to the limit of its soly. in the given concn. of AcOH. The thermostability of the product increases with the no. of formal groups and correspondingly decreasing no. of AcO, at about const. no. of OH. Presence of too high a no. of AcO lowers the temp. of softening. This can be remedied by treating the product with 0.5% NaOH at 90-95°; the product then begins to crumple at about 170-175° and becomes slightly yellowish at 200°. Products with  $\alpha = 65$  are sol. in  $\text{C}_6\text{H}_5\text{COCH}_3$ ; with  $\alpha$  below 78-80 they are sol. in alc., benzene (1:4), above 80 only in phenol, pyridine, and chloroform hydrocarbons. N. Thom

Vinyl ester of formic acid. S. N. Ushakov, I. A. Ardu-  
 zova, and E. N. Rostovskii. *Zhur. Priklad. Khim.* (J.  
 Applied Chem.) 20, 1013-18(1947).—Conditions have  
 been worked out for the successful catalytic synthesis by  
 $C_{11}H_7 + HCO_2H \rightarrow HCO_2CH_2C_{11}H_7$ , with max. suppression  
 of side reactions. Purified and dried  $C_{11}H_7$  mixed with  
 $HCO_2H$  vapor in the ratio  $C_{11}H_7$  15-17 mols. in  $HCO_2H$   
 1 mol. was fed over the catalyst with a time of contact  
 of 3.0-3.5 sec. The catalyst is  $(AcO)_2Zn$  or  $(HCO_2)_2Zn$   
 on activated C of grain size 1-2 mm., granulometric d.  
 0.81, apparent d. 0.837, true d. 1.88 g./cc., porosity  
 65.5%, sp. surface area 350 sq. m./g. (by sorption of  
 methylene blue). Under equal conditions, with  $HCO_2H$   
 Zn salt in the catalyst, reaction between lower temps. than the  
 starts and proceeds at considerably lower temps. than the  
 analogous reaction with  $AcOH$ ; thus, 65, 75, 85, and 95%  
 conversion of the acid were reached, with  $HCO_2H$  at  
 about 162, 168, 173, and 184°, with  $AcOH$  at 184, 189,  
 192, and 198°. The activity of the catalyst increases  
 with increasing amt. of Zn salt, e.g., with  $HCO_2H$ , amt.  
 of  $(HCO_2)_2Zn$  in the catalyst 9.4, 16.7, and 20%, a degree  
 of conversion of 80% was reached at about 173, 163,  
 and 160°, of 90% at, resp., 178, 160, and 168°. Detns.  
 of the amt. of ester in the condensate, with an 18% Zn  
 salt catalyst, at 180-195°, contact time 3.0-3.5 sec.,  
 $C_{11}H_7:HCO_2H = 10:1$  (molar), gave, for runs of 19-24,  
 24-30, and 40-45 hrs., resp., yields of 57, 54.1, and 61.5%  
 of  $HCO_2H$  passed, 74, 71, and 81% of  $HCO_2H$  spent.  
 In addn. to  $HCO_2C_{11}H_7$  and unreacted  $HCO_2H$ , the con-  
 densate contains about 5%  $AcH$ , evidently due to the side

reaction  $HCO_2C_{11}H_7 + HCO_2H \rightarrow 2CO + H_2O + AcH$ .  
 Polymerization of  $HCO_2C_{11}H_7$  in a sealed tube in the  
 presence of  $Bz_2O_2$ , 1.5 hrs. at 80°, gave 90% of solid  
 product insol. in  $EtOH$ ,  $C_{11}H_7$ ,  $CCl_4$ , and  $EtOAc$ , sol. in  
 $Me_2CO$ . The polymer formed on long standing, without  
 heating and without  $Bz_2O_2$ , was sol. in  $Me_2CO$  and  $CHCl_3$ ,  
 viscosity of a 0.2% soln. in  $Me_2CO$  0.352 centipoise, mol.  
 wt. 71000. This polyvinyl formate is readily saponi-  
 fied in boiling  $H_2O$  (96% in 6.5 hrs.), the  $HCO_2H$  formed  
 accelerating the reaction; pptn. with  $Me_2CO$  sepd. the  
 polyvinyl alc., white powder sol. in  $H_2O$ . The  $HCO_2$   
 $C_{11}H_7$  monomer is readily saponified with  $H_2O$ , with  $AcH$  as  
 a by-product. Thus, 25.6 g. of 90.9%  $HCO_2C_{11}H_7$   
 ( $HCO_2H$  1.6,  $AcH$  7.5%), boiled with 50 g.  $HCO_2H$   
 (80.5%), gave 11.8 g.  $AcH$  and 61.8 g. residue, fraction-  
 ated into b. 95-100, 100-103, and 103-105°, contg.,  
 resp., 81.7, 90.12, and 92.10%  $HCO_2H$ . N. Thoa

**Copolymerization of mixtures of vinyl esters with maleates.** I. Copolymerization of ethylene glycol maleate with vinyl formate. S. N. Ushakov and S. P. Mitzen-gender. *Zhur. Priklad. Khim.* (J. Applied Chem.) 20, 1261-9(1947).—Redistd. ethylene glycol was con-densed with an equimol. proportion of redistd. maleic acid according to the method of Kropa and Bradley (C.A. 34, 1199) or with a 20% excess of maleic acid according to the method of Rust (C.A. 34, 1773). The products were slightly colored resins of mol. wt. 2300-2800 and acid no. 20-25. These polyesters contain glycol maleate ester linkages (II). The polyesters were condensed with vinyl formate (III) with  $Bz_2O_3$  as a catalyst. The concns. studied were 50-330 parts II and 3 parts  $Bz_2O_3$  per thousand of total wt. The mixt. of polyester and II was held at 45° for 30-45 min. to obtain soln.  $Bz_2O_3$  was added and after 10-15 min. the soln. was warmed to 55° and allowed to polymerize for 10-18 hrs. The resulting copolymerizate (III) was freed from II polymer and unreacted polyester by long extr. with acetone in a Soxhlet flask. The percentage copolymerization was 00-07% for II: polyester ratios of 1:1 to 2:2.6 but decreased to 01% and 00% for ratios of 1:5 and 1:10, resp. Analysis for formate groups in III showed copolymerization to be the controlling reac-tion, since even with the max. concn. of II all the II was combined with the polyester. The product III was a three-dimensional polymer in which the basic structural

unit was  $-OCH_2CH_2OOCCH=CHCOOCH_2-$ . A modified



copolymer was made by sapon. the formate groups to form a polyester-vinylale. condensation product. Sapon. in even weak alk. solns. was undesirable because the I were broken under these conditions. III and  $H_2O$  were heated on a water bath with vigorous agitation to ac-complish the sapon. The aq. phase was replaced with fresh 11% every 12-15 hrs. to avoid the formation of suffi-cient acidity to cause hydrolysis of the I. The amt. of acid liberated was a linear function of time up to 70 hrs. No maleic acid was detected in the hydrolyzate after 37 hrs. After 50 hrs., 72% of the formate groups had been hydrolyzed to yield  $HCOOH$  and 11% of the I groups had been hydrolyzed to yield maleic acid. At 70 hrs. the per-centage of formate groups hydrolyzed reached the max. value of 82%. Thereafter hydrolysis occurred at the I ex-clusively and increased from 20% to 30% as the sapon. time increased from 70 to 90 hrs. Thus, splitting of I occurred more readily as the hydrophile nature of the polymer was increased by replacing the formate groups with hydroxyls.

H. K. Livingston



PA 47/49T16

USHAKOV, S. N.

USSR/Chemistry - Plastics  
Chemistry - Vinyl Alcohol

Nov 48

"Esterification of Polyvinyl Alcohol by Means of Dibasic Aliphatic Acids," S. N. Ushakov, R. K. Gavrilina, P. A. Medvedeva, Chair of Plastics Technol, Leningrad Tech Inst, 8 pp

"Zhur Priklad Khim" Vol XXI, No 11 1948

Shows that in the reaction of polyvinyl alcohol with chloroanhydride and polymer of adipic acid anhydride in pyridine, esters with varying degrees of esterization, depending on relation of original components, are formed.

47/49T16

USSR/Chemistry - Plastics (Contd)

Nov 48

Insoluble products are formed if more than 0.1 mole of chloroanhydride or adipic acid anhydride is added per mole of polyvinyl alcohol. If the reaction is conducted in a medium of concentrated acetic acid mixed complex esters, polyvinyl acetate adipates are derived. Shows that conditions of esterification of polyvinyl alcohol by succinic acid anhydrides are analogous to those of esterification by adipic acid anhydride. Submitted 1 Apr 48.

47/49T16

USHAKOV, S. N.

Ushakov, S. N., Gavurina, R. K. and Riadinskaia, N. M., On the homogeneity of the composition of polyvinylbutyrales obtained by methods of the homogeneous and heterogeneous acetalation. P. 1126.

The degree of physical and chemical homogeneity of polyvinylbutyrales, obtained by the homogenous and the heterogeneous methods of synthesis is approximately the same.

Chair of Technology of Plastic Masses  
Leningrad Technological Institute.  
April 1, 1948.

SO: Journal of Applied Chemistry (USSR) 21, No. 11 (1948)

USHAKOV, S. N.

Ushakov, S. N., Gavurina, R. K. and Tsubina, Kh. V. "On the dehydration of polyvinyl alcohol," In the Symposium: Investigations in the field of complex-molecular compounds, Moscow-Leningrad, 1949, p. 182-92, - Bibliog: 5 items

So: U-5241, 17 December 1953, (Letopis 'Zhurnal 'n-kh Statey, No. 26, 1949)

CA

21

Inst. Org. Chem., AS

Copolymerization of allyl alcohol and its derivatives with sulfur dioxide. S. N. Lashakov, I. A. Arbutova, and V. N. Hrenkova. *Izv. Akad. Nauk S.S.S.R., Otdel Khim. Nauk* 1949, 551-6. —Copolymerization of allyl alc. with  $\text{SO}_2$  at  $0-25^\circ$  in the presence of 0.02%  $\text{AgNO}_3$  in EtOH was investigated; the product, colorless amorphous solid after washing with Et<sub>2</sub>O and EtOH contained 25.65-29% S; in a 48-hr. reaction with 1:2 molar ratio at  $15^\circ$  with 0.2% catalyst, the yield rises rapidly from 20% in 18 hrs. to over 80% in 30 hrs. and is almost 100% in 48 hrs.; the temp.-yield curve is linear; the effectiveness of the catalyst is displayed almost identically in 0.05-0.2% concns., but lower amts. give sharply decreased yields; increase of  $\text{SO}_2$  ratio to 8 gives a 97.8% yield, against 89.7% in a 0-day polymerization at  $9^\circ$  with 0.02% catalyst. The polysulfone products are unstable to hot aq. alkalis (even 2%), swell in water, and hydrolyze slowly on boiling; they are sol. only in pyridine and can be reprecipitated from solns. in strong mineral acids without change. Viscosities of 0.2% samples made with 0.003-0.04% catalyst were closely grouped at 1.153-1.236 centipoises and essentially no fractionation was achieved by stepwise pptn. from sirupy  $\text{H}_3\text{PO}_4$  by  $\text{H}_2\text{O}$  (as dil.  $\text{H}_3\text{PO}_4$ ). Treatment of the product with paraformaldehyde (0.73-1.0 g. per 2 g. polymer) in concd. HCl at  $35-40^\circ$  for 4 hrs. gave an amorphous ppt., after the gelation stage, which contained 170-2% formal group substitution; the products are insol. in all org. solvents and slowly dissolve in HCl with loss of  $\text{CH}_2\text{O}$ . Similar copolymerization of diallyl formal and diallyl acetal with  $\text{SO}_2$ , with  $\text{AgNO}_3$  catalyst (dissolved in allyl acetate) for 3 hrs. to 3 days (without catalyst), gave 38% copolymer with the formal and 43-49% polymer with the acetal when  $\text{AgNO}_3$  was used (12% without catalyst in 3 days); the acetal formed the copolymer vigorously even at  $-15^\circ$ , but the action slows down very rapidly; the products (empirical formula for the formal being  $\text{C}_{11}\text{H}_{13}\text{S}_2\text{O}_4$  and for acetal  $\text{C}_{11}\text{H}_{13}\text{S}_2\text{O}_6$ ) are insol. in org. solvents and are sol. slowly in HCl with aldehyde evolution. G. M. K.

10

CA

Synthesis of vinyl ethers of polyvinyl alcohol S. N. Shakov and S. I. Kirillova. *Zhur. Priklad. Khim.* (J. Applied Chem.) 22, 1004 (1969) (1969). *Vinylization of polyvinyl alc.* is best done in aq. alk. soln. at 12-18 atm. of  $C_2H_4$  at 100-15°. At 128-30° the main reaction is mere dehydration of the alc. After 10-20 hrs. reaction there are formed products, sol. in  $H_2O$ , MeOH, and 80% EtOH, which contain 3.6-6.0 mol-% vinyl groups. The yields range from 50 to 70%. The products are yellowish to brown solids; lighter products are secured by using 8% KOH with addn. of  $NH_4OH$  and  $ZnO$ ;  $R_2N$  or quinaldine gave poor results leading to insol. products. The detn. of the vinyl groups was done by detn. of AcH following hydrolysis with dil.  $H_2SO_4$ . G. M. Kosolapoff

C. ★  
 Synthesis of *m*-hydroxyzone. N. N. Vishokov and K. V. Frankelberg (Polymer. Plast. Inst., Leningrad). *Izv. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk* 1950, 204-76. Addn. of 100 g. H<sub>2</sub>SO<sub>4</sub> to 110 g. KNO<sub>3</sub> in 325 g. concd. H<sub>2</sub>SO<sub>4</sub> at 5° over 4-5 hrs. and diln. with H<sub>2</sub>O gives 40-41% *m*-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CHO, m. 64°, along with some 25% *o*-isomer which remains in the mother liquor. At 25-35° the yield of *m*-isomer is raised to 75-80%. Addn. of the product with stirring to SnCl<sub>4</sub> in HCl with cooling (spontaneous rise of temp. to 60-80° occurs in spite of cooling), followed by diazotization at 2° with NaNO<sub>2</sub>, addn. of KI soln. at 5°, melting stand 12 hrs., and heating 1 hr. to 80-90° gave *m*-IC<sub>6</sub>H<sub>4</sub>CHO. The use of a very high proportion of HCl (40 mols.) gives poorer results (15-20% yield) than 8 moles HCl (theoretical amt.), which gave 28%, or 10-11 moles HCl (30-41% yield); the product, isolated by steam distn., m. 66-7°. The reaction of this with MeMgI conducted as usual in Et<sub>2</sub>O gave 70% *m*-IC<sub>6</sub>H<sub>4</sub>CH(OH)Me, b<sub>p</sub> 119-20°, d<sub>4</sub><sup>20</sup> 1.707, n<sub>D</sub><sup>20</sup> 1.6215. Dehydration of this by passage over Al<sub>2</sub>O<sub>3</sub> at 300° proceeds well for 15-20 min. with formation of 98% pure product, after which period decompn. begins and iodine is evolved; the same occurs at 260-70°. Distn. of the carbinol with KHSO<sub>4</sub> in the presence of hydroquinone (0.1-0.2%) at 190-200° and 50-60 mm. in a CO<sub>2</sub> atm. gave a 71-4% yield with 13-14% (by wt.) of KHSO<sub>4</sub>; a smaller ratio of KHSO<sub>4</sub> gave lower yields. The pure *m*-IC<sub>6</sub>H<sub>4</sub>CH:CH<sub>2</sub>, b<sub>p</sub> 60-71°, b<sub>p</sub> 71-3°, d<sub>4</sub><sup>20</sup> 1.074, n<sub>D</sub><sup>20</sup> 1.6390, polymerizes with 0.1% Bz<sub>2</sub>O<sub>2</sub> in 1-2 hrs. at 80-100° to a clear solid polymer, d<sub>4</sub><sup>20</sup> 1.80, n<sub>D</sub><sup>20</sup> 1.6450 (highest among org. polymers).  
 G. M. Kosolauoff

1ST AND 2ND CODES																										3RD AND 4TH CODES																									
PROCESSING AND PROPERTIES INDEX																																																			
8																										29																									
<p><b>Contemporary State of the Theory of Copolymerization.</b> (In Russian.) S. N. Lyubkov, S. P. Mitsengendler, and G. A. Shtraukhman. <i>Uspekhi Khimii</i> (Progress in Chemistry), v. 19, May-June 1950, p. 265-291.</p> <p>Thoroughly reviews the above, including derivation of the basic equation of copolymerization, determination of copolymerization constants, influence of foreign substances and temperature, relative activity of monomers with free radicals, influence of extent of transformation on composition of polymers, etc. 48 ref.</p>																																																			
<p>ASAC-SLA METALLURGICAL LITERATURE CLASSIFICATION</p>																																																			
REGIONAL INDEX																										REGIONAL INDEX																									
SUBJECT INDEX																										SUBJECT INDEX																									

**B**

Copolymerization of Vinylacetate and Monomethylmaleate. (In Russian.) S. N. Ushakov, S. P. Mitsengendler, and B. M. Polyatskina. *Zhurnal Prikladnoi Khimii* (Journal of Applied Chemistry), v. 23, May 1950, p. 512-520.

Results of experimental investigation are tabulated and charted. Mechanism and kinetics are discussed.  
11 ref



Application of the Equation of Copolymerization for Calculation of Composition and Structure of Copolymers. (In Russian.) S. N. Ushakov, S. P. Mitsengol'er, and B. M. Polyutskina. *Zhurnal Prikladnoi Khimii* (Journal of Applied Chemistry), v. 23, May 1950, p. 521-529.

Above was investigated in an attempt to explain the influence of extent of transformation on composition of the copolymers of vinylacetate and maleic ester (see above abstract), also to determine the composition of the copolymers by using the constants of copolymerization. Experimental data confirm validity of the calculations. Method of investigation is described. Data are tabulated and charted.

177128

USSR/Chemistry - Synthetic Resins and  
Elastomers

Mar 51

"Effect of Solvents and Temperature on the Copoly-  
merization Constants of Vinylacetate and Monomethyl-  
maleate," S. N. Ushakov, S. P. Mitsengendler, B. M.  
Polyatskina, Chair of Plastics, Leningrad Tech Inst  
Imeni Lensovet

"Zhur Prikl Khim" Vol XXIV, No 3, pp 289-295

Exam effects of solvents and temp on const  $\sigma$  and  $\mu$   
of copolymerization of vinylacetate and monomethyl-  
maleate. Found no change of const in presence of

177128

USSR/Chemistry - Synthetic Resins and  
Elastomers (Contd)

Mar 51

solvents. As to temp, found both const approximately  
doubled between 56 and 78°C, but temp actually had  
small effect on compn of copolymers.

177128

USHAKOV, S. N.

CA

31

The effect of solvents and temperature on the copolymerization constants of vinyl acetate and monomethyl maleate. V. S. N. Ushakov, S. P. Mitsengendler, and B. M. Polyatskina (Leningrad Technol. Inst., Leningrad). *J. Applied Chem. U.S.S.R.* 24, 319-25(1951) (Engl. translation).—See *C.A.* 40, 774c. R. M. S.

1952

USHAKOV, S. N.

USSR/Chemistry - Plastics

May 51

"Copolymerization of Vinyl Acetate and Maleic Anhydride and the Properties of the Copolymers Obtained," S. N. Ushakov, S. P. Mitsengendler, V. A. Chekhovskaya, Chair of Plastics, Leningrad Tech Inst

"Zhur Prikl Khim" Vol XXIV, No 5, pp 485-489

Same relationships are observed in copolymerization of vinyl acetate with maleic anhydride as in the case of vinyl acetate and monomethyl maleate. Maleic anhydride, like its ester, can

183T36

USSR/Chemistry - Plastics (Contd)

May 51

combine with itself in presence of complex vinyl esters to yield copolymers contd higher than 50% maleic component. Obtained copolymers are easily sapon with H<sub>2</sub>O to form high-mol compds with side rings on chain (lactones).

183T36

USHAKOV, S.N.; MITSENGENDLER, S.P.; POLYATSKINA, B.M.

Application of newer methods of study to copolymerization of vinyl acetate with the maleates. Khim. i Fiz. Khim. Vysokomolekul. Soedineniy, Doklady 7-oy Konf. Vysokomolekul. Soedineniyam '52, 19-27. (MLRA 5:7) (CA 47 no.15:7820 '53)

USSR/Chemistry - Plastics

Jan 52

"Hydroxyallyl Ethers of Cellulose and Their Co-polymerization With Sulfurous Anhydride," S. N. Ushakov, O. M. Klimova

"Zhur Prih Khim" Vol XXVI, No 1, pp 46-56

Uniform low-mol allyl ethers of cellulose were synthesized by homogeneous medium method in following manner. Hydroxyethyl ether of cellulose (I) (sol in aq alkali solns of 18-20% concn) was prepd. I reacted with allylbromide in different proportions to yield hydroxyethylallyl ethers of cellulose (III) with different degs of substitution. Under

206741

USSR/Chemistry - Plastics (Contd)

Jan 52

exposure to air III was oxidized into insol product (IV). Polymerization of III under action of benzoyl peroxide and heat stopped at stage of gel-formation, due to spatial hindrances.

206741

USHAKOV, S. N.

USHAKOV, S.N.

B. T. R.  
Vol. 3 No. 4  
Apr. 1954  
Chemistry-Organic

4617\* Alkali Derivatives of Polyvinyl Alcohol. (Russian.)  
S. N. Ushakov and E. M. Lavrenteva. *Zhurnal Prikladnoi Khimii*, v. 26, no. 9, Sept. 1953, p. 960-968.  
Describes action of aqueous solutions of caustic soda. Product is analogous to the alkali derivative of cellulose. Tables, graph, photograph. 7 ref.

3.  
② chem

MF  
7-28-54

USHAKOV, S. N.

USSR/Chemistry      Synthesis processes

Card                : 1/1      Pub. 40- 18/27

Authors            : Ushakov, S. N., and Solomon, O. F.

Title              : About the synthesis of cyclooctatetraene

Periodical        : Izv. AN SSSR. Otd. khim. nauk 4, 694 - 706, July - August 1954

Abstract          : The effect of various factors on the polymerization reaction of acetylene yield and rate of formation of cyclooctatetraene during the process of catalytic polymerization of acetylene under pressure, was investigated. Polymerization of acetylene into cyclooctatetraene offers best results in the absence of ethylene oxide and calcium carbide. Water traces and some unidentified foreign admixtures contained in the catalyst, were found to be the only inhibitors of the polymerization reaction. The effect of pressure in the reaction vessel on the yield of cyclooctatetraene, is explained. Twenty-four references: 1 USSR; 10 German; 2 English; 11 USA (1911 - 1952). Tables; graphs.

Institution : The Leningrad Technological Institute, Leningrad

Submitted    : July 6, 1953





USHAKOV, S.N.

USSR/ Chemistry - Synthetics

Card 1/1 Pub. 40 - 15/2'

Authors : Ushakov, S. N., and Kononova, T. A.

Title : ~~SYNTHESIS OF POLYVINYL ALCOHOL ESTERS~~  
Synthesis of polyvinyl alcohol esters

Periodical : Izv. AN SSSR. Otd. khim. nauk 1, 117-125, Jan-Feb 1955

Abstract : Experimental data are given on the development and improvement of methods for the synthesis of polyvinyl alcohol esters (polyvinylformate, polyvinyl acetate, polyvinylpropionate, polyvinylbutyrate and polyvinylisobutyrate) containing various amounts of free hydroxyl groups and having uniform average length of the macromolecular chain and polydispersion. The results obtained with the aid of the new methods are described. Thirteen references: 4 USSR, 4 German, 2 USA and 3 English (1926-1949). Tables; graph.

Institution : The Leningrad Technological Inst. Leningrad

Submitted : April 23, 1954

USHAKOV, S. N.

USSR/ Chemistry - Chemical technology

Card 1/1 Pub. 40 - 19/26

Authors : Ushakov, S. N., and Kononova, T. A.

Title : About certain physico-chemical properties of polyvinyl alcohol esters

Periodical : Izv. AN SSSR. Otd. khim. nauk 2, 335 - 343, Mar-Apr 1955

Abstract : Tests were made to determine the vitrification temperatures and mechanical properties of complete polyvinyl alcohol esters and formic, propionic, n-butyric and isobutyric acids and a series of products obtained through their partial saponification. The vitrification points were found to be constant up to a free hydroxyl content of 30 mol/%; they increase in proportion to the drop in ester group content. The anomalous change in the vitrification point of formic esters of polyvinyl alcohol is explained. The strength, modulus and elongation of polyvinyl alcohol ester films were determined in vitreous and high-elastic states. Nine references: 8 USSR and 1 German (1939-1955). Tables; diagrams.

Institution : The Leningrad Soviet Technological Institute, Leningrad

Submitted : April 23, 1954

USSR/ Chemistry - Polymerization

Card 1/1 Pub. 40 - 15/25

Authors : Ushakov, S. N., and Nikolayev, A. F.

Title : Polymerization and copolymerization of N-vinyl compounds. Part 1. Copolymerization of vinyl carbazole with vinyl esters

Periodical : Izv. AN SSSR. Otd. khim. nauk 1, 83-91, Jan 1956

Abstract : New hitherto unknown vinyl carbazole and vinyl ester copolymers of organic acids (formic, acetic, propionic and butyric) obtained through mass polymerization are described. The causes for the reduction in the rate of vinyl ester copolymerization followed by an increase in the length of the acid residue chain of vinyl ether are explained. The copolymerization constants were established for several vinyl base compounds and the differential and integral compositions of the vinylcarbazole copolymers were estimated. Thirty-six references: 17 USA, 6 USSR, 2 Germ., 1 French and 10 Eng. (1937-1953). Tables; graphs.

Institution : Leningrad Technological Institute im. Leningrad Soviet

Submitted : March 10, 1955

USHAKOV, S. N.  
USSR/Chemistry of High-Molecular Substances, F

Abst Journal: Referat Zhur - Khimiya, No 19, 1956, 61745

Author: Ushakov, S. N., Nikolayev, A. F.

Institution: None

Title: Polymerization and Copolymerization of N-vinyl Compounds. Communication 2. On Some Characteristics of the Reaction of Copolymerization of Vinyl Acetate and Vinyl Carbazole and the Properties of the Copolymers

Original  
Periodical: Izv. AN SSSR, otd. khim. n., 1956, No 2, 226-231

Abstract: Rate of copolymerization of vinyl carbazole (I) and vinyl acetate (II) (temperature 80° and 100°, initiator benzoyl peroxide) passes through a minimum at a concentration of I of 10-20 mol %. At 65° and a 10-35% concentration of I polymerization does not take place. Rate of copolymerization of I and II is proportional to the square root of the concentration of the initiator and the higher the concentration of I in the mixture the higher is the rate of

Cs

Card 1/2

"APPROVED FOR RELEASE: 03/14/2001

CIA-RDP86-00513R001858120016-9

Phthalic esters of erythritol  
Klimova and

APPROVED FOR RELEASE: 03/14/2001

CIA-RDP86-00513R001858120016-9"

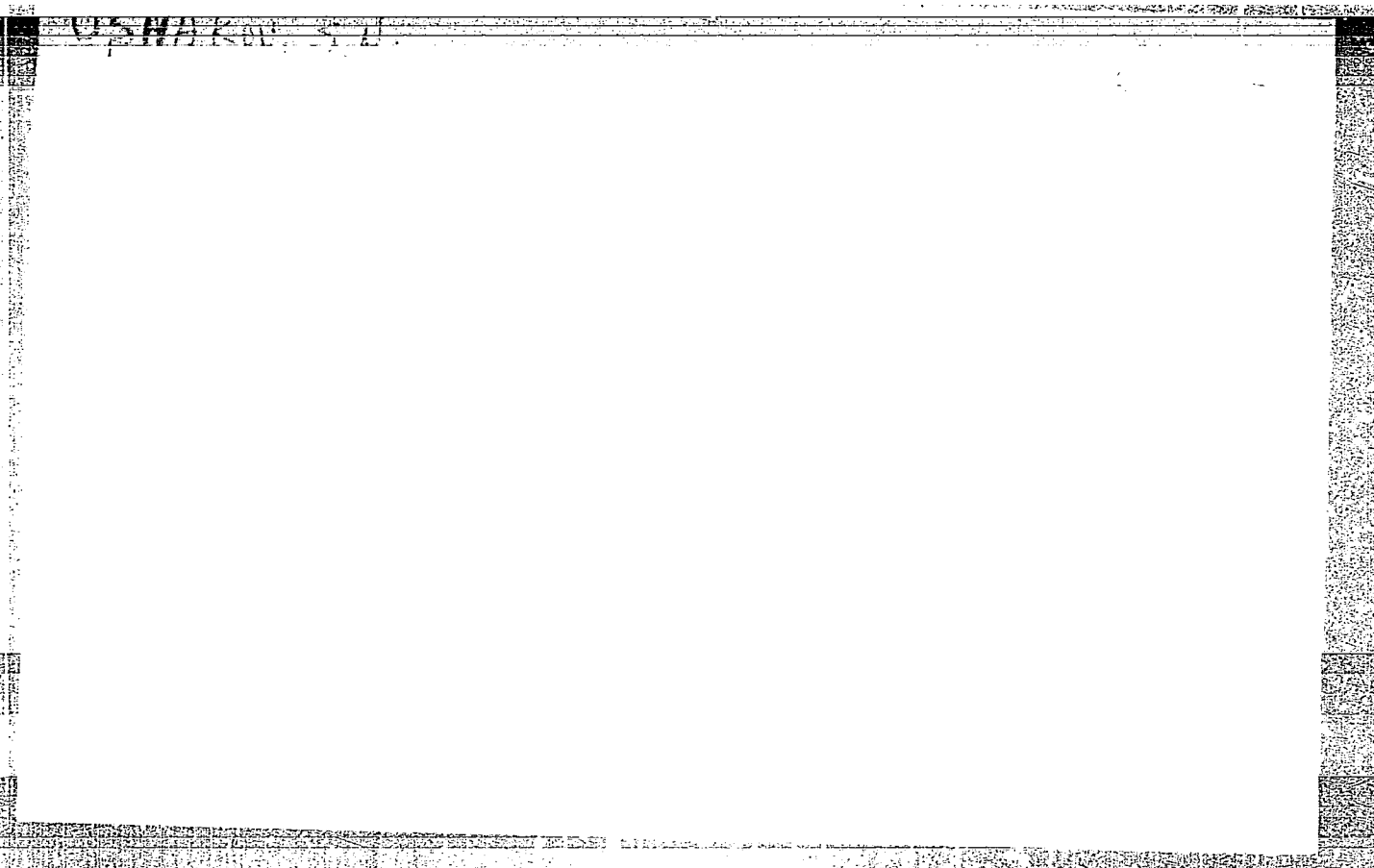
USHAKOV, S. N., and LAURENTYeva, E. N.

"A few new polymerx derived from polyvinylalcohol," a paper  
presented at the 9th Congress on the Chemistry and Physics of High Polymers,  
28 Jan-2 Feb 57, Moscow, Polymer Research Inst.

B-3,004,395

"APPROVED FOR RELEASE: 03/14/2001

CIA-RDP86-00513R001858120016-9



APPROVED FOR RELEASE: 03/14/2001

CIA-RDP86-00513R001858120016-9"



"APPROVED FOR RELEASE: 03/14/2001

CIA-RDP86-00513R001858120016-9

ISHAZOV S.N.

APPROVED FOR RELEASE: 03/14/2001

CIA-RDP86-00513R001858120016-9"

...  $\text{Na}_2\text{P}_2\text{O}_7$  and glucose was examined, with and without  $\text{CCl}_4$  in the system. The action of  $\text{CCl}_4$  is sup-  
posed to be under these conditions and the reaction is sup-  
posed to be radical in nature.

7.5E4

USHAKOV, S.N.; MITSENGENDLER, S.P.; KRASULINA, V.N.

Copolymerization of diethylene hydrocarbons with vinylalkyl ethers.  
Report No.2: Copolymerization of divinyl with vinylalkyl ethers  
in emulsion at low temperatures. Izv.AN SSSR Otd.khim.nauk no.4:490-493  
Ap '57. (MIRA 10:11)

1. Institut vysokomolekulyarnykh soyedineniy AN SSSR.  
(Polymerization) (Ethers)

Hark S.S.S.R., Odes. Kama, Mark 1957, (66-8/1957).  
 Copolymers of  $\text{CH}_2\text{ClCH}(\text{CH}_3)\text{CH}_2$  (I) with  $\text{RCO}_2\text{CH}(\text{CH}_3)\text{CH}_2$ ,  
 $\text{AcOCH}(\text{CH}_3)\text{CH}_2$ ,  $\text{EtCOCH}(\text{CH}_3)\text{CH}_2$ , and  $\text{PrCOCH}(\text{CH}_3)\text{CH}_2$ , were  
 prepd at  $65^\circ$  with 2-2%  $\text{SnO}_2$  initiator. The copolymers

[illegible]

USHAKOV, S.N

USHAKOV, S.N.; TRUKHMANOVA, L.B.

Copolymerization of chloroprene and vinyl esters. Report No.2:  
"Copolymerization Limit" and rates of reactions during the  
copolymerization of chloroprene with vinyl esters. Izv. AN SSSR.  
Otd. khim. nauk no.9:1072-1079 S '57. (MIRA 10:12)

1. Institut vysokomolekulyarnykh soyedineniy AN SSSR.  
(Polymerization) (Chloroprene) (Vinyl alcohol)

1/21/64 5:10 PM

[illegible]

52-12-6/20

AUTHORS: Ushakov, S.N., Ivanov, S.S.

TITLE: On the Co-Polymerization of Divinyl With Vinyl Formate  
(O sopolimerizatsii diviniila s vinilformiatom).

PERIODICAL: Izvestiya AN SSSR Otdeleniye Khimicheskikh Nauk, 1957, Nr 12,  
pp. 1465-1471 (USSR)

ABSTRACT: The co-polymerization of divinyl with complicated vinyl ethers is of interest because of the possibility thus arising of modifying the properties of vinyl polymers. Because of the low velocities of co-polymerization reaction in the medium of hydrocarbon, and in view of the possibility of hydrolysis in emulsion, it has hitherto been considered impossible to obtain divinyl co-polymers with complicated vinyl ethers. In this paper the authors speak about co-polymerization, which has hitherto not been described in publications dealing with this field. The conditions of the co-polymerization of these monomers in the mass in the presence of the oxidation regeneration system (okislitel'novosstanovitel'naya sistema) is described. The influence exercised by the nature of the radical (bound to iron) upon the velocity of co-polymerization and the yield of co-polymers was described. The use of iron stearate (instead of naphtenate) increases the degree

Card 1/2

62-12-6/20

# On the Co-Polymerization of Divinyl With Vinyl Formate

of conversion nearly five-fold. In the fractionation of the co-polymer the fractions are distinguished by their molecular weight and not by their chemical structure. Furthermore, the possibility of the saponification of the formyl groups of the co-polymer was found to exist, and the influence exercised by the free hydroxyl groups upon some properties of the polymers obtained is described. Finally, the constants of the co-polymerization of divinyl with vinyl formate was uniquely determined. There are 6 tables, and 15 references, 9 of which are Slavic.

ASSOCIATION: Institute for High-Molecular Compounds AN USSR (Institut vysokomolekulyarnykh soedineniy Akademii nauk SSSR).

SUBMITTED: July 9. 1956

AVAILABLE: Library of Congress

Card 2/2 1. Divinyl-Co-Polymerization 2. Vinyl-Co-Polymerization



"APPROVED FOR RELEASE: 03/14/2001

CIA-RDP86-00513R001858120016-9

APPROVED FOR RELEASE: 03/14/2001

CIA-RDP86-00513R001858120016-9"

AUTHORS:

USHAKOV S. N.

Rostovskiy, Ye. M., Ushakov, S. N., Barinova, A. M.

62-1-10/29

TITLE:

On the Properties of a Series of Complex Vinyl Ethers (O svoystvakh ryada slozhnykh vinilovykh efirov)  
Report 1: On the Polymerization and Velocity of the Saponification of the Monomers (Soobshcheniye 1. O polimerizatsii i skorosti omyleniya monomerov)

PERIODICAL:

Izvestiya AN SSSR Otdeleniye Khimicheskikh Nauk, 1958,  
Nr 1, pp 59 - 63 (USSR)

ABSTRACT:

In the hitherto published reports one was restricted to mainly the data about the boiling temperatures and some other physical constants of the monomers. Only in some papers (ref. 1,3,4) the properties of the polymers of complex vinyl ethers were investigated more precisely. The present report deals with the kinetics of the polymerization of a series of complex vinyl ethers, as well as with the detection of their saponification velocity, and with the temperatures of the vitrification of polymers (tables 1,2). The polymerization in the mass as well as the velocity of the saponification of several complex vinyl ethers, and the temperature of the vitrification of polymers were investigated. Furthermore the structure of the azyradicals and their influence on the initial velocity of the polymerization and kinetics of

Card 1/2

On the Properties of a Series of Complex Vinyl Ethers  
 Report 1: On the Polymerization and Velocity of the Saponification of the  
 Monomers

62-1-10/2)

the hydrolysis of these ethers were precisely detected. It was  
 also explained that the influence of the size and the structure  
 of the accessory groups of the polymers on the temperatures of  
 the vitrification has a similar character in the series of com-  
 plex vinyl ethers, acrylates, and metacrylates. There are  
 2 figures, 2 tables, 23 references, 11 of which are Slavic.

ASSOCIATION:

Institute of High-Molecular Compounds, AS USSR (Institut  
 vysokomolekulyarnykh soedineniy Akademii nauk SSSR).

SUBMITTED:

August 25, 1956

AVAILABLE:

Library of Congress

1. Complex vinyl ethers-Properties
2. Complex vinyl ethers-  
Polymerization
3. Complex vinyl ethers-Saponification-Velocity

Card 2/2

AUTHORS:

SOV/62-58-8-9/22  
Nikolayev, A. F., Ushakov, S. N., Rozenberg, M. E.

TITLE:

Polymerization and Co-Polymerization of n-Vinyl Compounds  
(Polimerizatsiya i sopolimerizatsiya n-vinil'nykh soyedineniy)  
Note 4: The Polymerization of Vinyl Phthalimide (Sobshcheniye  
4. Polimerizatsiya vinilftalimida)

PERIODICAL:

Izvestiya Akademii nauk SSSR, Otdeleniye khimicheskikh nauk,  
1958; Nr 8, pp. 968-972 (USSR)

ABSTRACT:

In publications there exist few reports on the polymerization of vinyl phthalimide. In the introduction the first experiments and the preliminary work for the production of polyvinyl phthalimide and vinyl phthalimide are discussed in short (Refs 1-4). In the present paper the authors describe the polymerization of vinyl phthalimide (in block and in the solvent). Also data on the properties of the polymer are given. The dependence of the polymerization rate of vinyl phthalimide and of the molecular weight of the polymer on the conditions of the polymerization in the presence of benzoyl peroxide and azobisisobutyro nitrile were characterized. It was found that powdery polyvinyl phthalimide produced in the polymerization

Card 1/2

SOV/62-58-8 9/22

Polymerization and Co-Polymerization of  $\alpha$ -Vinyl Compounds. Note 4: The  
Polymerization of Vinyl Phthalimide

of the monomer in benzene is also suited for the further processing. The polymer obtained has enough hardness and heat resistance, and is soluble to a limited extent in organic substances. There are 3 figures, 5 tables, and 12 references, 3 of which are Soviet.

ASSOCIATION: Leningradskiy tekhnologicheskii institut im. Lensovet  
(Leningrad Technological Institute imeni Lensovet)

SUBMITTED: January 14, 1957

Card 2/2

USHAKOV, S.N., ~~sasluzhennyy~~ *sasluzhennyy* *deyatel' nauki i tekhniki* RSFSR, prof.;  
~~LEVENT'YEVA, Ye.M., mladshiy nauchnyy sotrudnik.~~

Growth of synthetic fiber production. Tekst. prom. 18 no. 3: 51-52  
Mr '58. (MIRA 11:3)

1. Chlen-korrespondent Akademii nauk SSSR (for Ushakov)  
(Textile fibers, Synthetic)

79-28-5-33/69

AUTHORS: Arbuzova, I. A., Ushakov, S. M., Plotkina, S. A., Yefremova,  
V. M., Ulezlo, I. K.

TITLE: On the Conversion Reactions of Methylolmetacrylamide ( O  
reaktsiyakh prevrashcheniya metilolmetakrilamida)

PERIODICAL: Zhurnal Obshchey Khimii, 1958, Vol 28, Nr 5,  
pp. 1266 - 1269 (USSR)

ABSTRACT: In carrying out one of the experiments for the synthesis of  
methylolmetacrylamide according to Feuer, Lynch ( Fayer i  
Linch) (Reference 1) the authors separated, besides this com-  
pound, also a product with the melting point 80.5 - 81.5°C  
which, until now, has not been identified as dimetacrylamido-  
dimethylether. Many experiments to isolate this product from  
the mixture of final products of the above synthesis did not  
succeed, which also was the reason for investigating the con-  
version reaction of methylolmetacrylamide more in detail. The  
experiments to realize the dimetacrylamidodimethylether by  
conversion of the methylolmetacrylamide with benzoylchloride

Card 1/3

On the Conversion Reactions of Methylolmetacrylamide

79-28-5-33/69

in alkaline medium according to Ziegner (Tsigezner) (Reference 3) did not succeed. Being of the opinion that the ether would have to form as a final product in the synthesis of methylenedimacrylamide in the presence of acidous catalysts the behaviour of methylolmetacrylamide in the presence of acidous catalysts was investigated. On heating of the latter with a small amount of hydrochloric acid it could be converted into the dimetacrylamidodimethylether. In the case of increased concentration this ether was converted to the already known methylenedimacrylamide (see reaction scheme). According to the data by Peter and Lynch, the methylolmetacrylamide polymerizes on heating in the presence of mineral acids and boron chloride ( $BCl_3$ ) with formation of unmeltable and insoluble polymers, which fact indicates a three-dimensional structure. The experiments carried out by the authors showed that the methylolmetacrylamide also polymerizes on the action of peroxide stimulators in which case polymers of a line or three-dimensional structure can be obtained, depending on the prevailing conditions. In the case of irradiation of this amide with ultraviolet light

Card 2/3



On the Conversion Reactions of Methylolmetacrylamide

79-28-5-33/69

a solid unmeltable polymer results from it. In the masspolymerization in the presence of benzoylperoxide a vitreous polymer forms which is insoluble in water and usual organic solvents. There are 6 references, <sup>none</sup> of which are Soviet.

ASSOCIATION: Institut vysokomolekulyarnykh soyedineniy Akademii nauk SSSR  
(Institute for High-Molecular Compounds, AS USSR)

SUBMITTED: April 29, 1957

Card 3/3

USHAKOV, S.N.; LEVRENT'YEVA, Ye.M.

Synthesis of vinyl acetate copolymers with crotonic acid and its derivatives. Zhur.prikl.khim. 31 no.11:1686-1691 N '58, (MIRA 12:2)

(Vinyl acetate)

(Crotonic acid)

5(3)

AUTHORS:

SOV/62-59-1-15/38  
Ushakov, S. N., Lavrent'yeva, Ye. M., Podgorskaya, K. S.

TITLE:

On the Synthesis of Methylol Croton Amide (O sinteze metilol-krotonamida)

PERIODICAL:

Izvestiya Akademii nauk SSSR, Otdeleniye khimicheskikh nauk, 1959, Nr 1, pp 91 - 94 (USSR)

ABSTRACT:

There are no data available in publications on the synthesis of methylol croton amide. In the present paper it was obtained by the authors according to the following scheme: crotonic acid  $\rightarrow$  crotonic acid chloride  $\rightarrow$  crotonic acid amide  $\rightarrow$  methylol croton amide. Crotonic acid was synthesized from malonic acid by the interaction with acetaldehyde in pyridine and with ethyl alcohol as a solvent. Crotonyl chloride was obtained by the effect of thionyl chloride on solid crotonic acid. Its yield amounted to 70% instead of 37% as mentioned in publications. There are numerous data on the synthesis of croton amide (Refs 4-9). It was obtained most easily by the effect of crotonyl chloride on liquid ammonia in ether solution at  $-35^{\circ}$ . Methylol croton amide

Card 1/3

SOV/62-59-1-15/38

# On the Synthesis of Methylol Croton Amide

was synthesized by the interaction of croton amide with paraform in the presence of sodium ethylate as a catalyst. Table 1 gives some data on some experiments of methylol croton amide synthesis. Methylol croton amide represents needle-shaped crystals which at low temperature are easily dissolved in water, alcohol and dioxane, and on heating in ethyl acetate, vinyl acetate and benzene. It was found that methylol croton amide can form ether on heating without a catalyst. The ether was obtained by heating methylol croton amide in toluene and distilling off the reaction water with the vapors of the solvent in the absence of the catalyst (Table 2). As may be seen from the analysis, the amount of nitrogen in ether approaches the theoretical content, and the melting point increased from 87° for methylol croton amide up to 136° for ether. The ether of methylol croton amide represents needle-shaped lustrous crystals which at low temperature are soluble in acetic acid and on heating in water, dioxane, benzene and xylene. There are 2 tables and 11 references, 1 of which is Soviet.

Card 2/3

On the Synthesis of Methylol Croton Amide

SOV/62-59-1-15/38

ASSOCIATION:

Institut vysokomolekulyarnykh soyedineniy Akademii nauk SSSR  
(Institute of High-Molecular Compounds of the Academy of  
Sciences, USSR)

SUBMITTED:

April 27, 1957

Card 3/3

USHAKOV, S.N.; LAVRENT'YEVA, Ye.M.; GEYSBERG, S.M.; SHEMKOV, N.K.

Synthetic fibers from polyvinyl alcohols. Khim.volok. no.4:  
3-5 '59. (MIRA 13:2)

1. Institut vysokomolekulyarnykh soyedineniy AN SSSR i Leningrad-  
skiy zavod.  
(Textile fibers, Synthetic) (Vinyl alcohol)

5 (3)

AUTHORS:

Ushakov, S. M., Lavrent'yeva, Ye. M.,  
Podgorskaya, K. S.

SOV/62-59-5-18/40

TITLE:

On the Synthesis of Methylene Biscrotonamide (O sinteze metilen-bis-krotonamida)

PERIODICAL:

Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk,  
1959, Nr 5, pp 888-891 (USSR)

ABSTRACT:

There are no references in publications with regard to the synthesis mentioned in the title. These compounds are interesting: they contain nitrogen, two double bonds, and are capable of copolymerization. I. A. Arbuzova carried out tests with acryl derivatives at the authors' institute. In this work, methylene biscrotonamide was synthesized in three different ways: 1) two molecules of methylol crotonamide separate water and formaldehyde, 2) the amide of crotonic acid separates water under the effect of methylol crotonamide, 3) the di-ester of methylol crotonamide separates formaldehyde with thermal treatment. The first reaction took place without catalyst by heating a xylene solution of methylol crotonamide. Table 1 shows data of this synthesis. Nitrogen content, double bonds, melting temperature, molecular weight and solubility of

Card 1/2

On the Synthesis of Methylene Biscrotonamides

SCV/62-59-5-13/40

the compound obtained were determined. (Data on analysis in table 2). The second reaction too, took place in xylene, with heating and without a catalyst. Tables 3 and 4 contain the same determinations of substances synthesized in the second way as table 2. Tables 5 and 6 show the corresponding data of the third way of synthesis. In this case the reaction desired was brought about by heating the di-ester in various aromatic solvents. The bromine number of the methylene biscrotonamide of the last two compounds obtained was close to the theoretically determined value. There are 6 tables.

ASSOCIATION: Institut vysokomolekulyarnykh soedineniy Akademii nauk USSR  
(Institute of High-molecular Compounds of the Academy of Sciences, USSR)

SUBMITTED: August 13, 1957

Card 2/2



5 (3)  
AUTHORS:

Nikolayev, A. F., Ushakov, S. N.,  
Krasnosel'skaya, I. G.

SOV/62-59-9-17/40

TITLE:

Polymerization and Copolymerization of N-Vinyl Compounds.  
Communication 5. Polymerization of Vinyl Succinimide

PERIODICAL:

Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk,  
1959, Nr 9, pp 1627 - 1630 (USSR)

ABSTRACT:

The present article describes the polymerization of N-vinyl succinimide (VS), which has not been previously described, and the properties of the polymers obtained are investigated. VS was prepared by a method described by the authors in reference 1, by pyrolysis from  $\beta$ -acetoxyethyl succinimide. The polymerization of VS succeeded only by using peroxide initiators. The polymerization was carried out at 50, 65, and 85° with 0.2% benzoyl peroxide (BP) in solid state and in solution. Figure 1 illustrates the influence of the temperature and figure 2 the influence of the concentration of the initiator on the polymerization rate. At 50° a maximum yield (98%) was obtained during 6 hours. The yield decreased with increasing temperature, but the reaction rate increased. The complete consumption of the monomer ended the polymerization. The polymer obtained is colorless, trans-

Card 1/2

Polymerization and Copolymerization of N-Vinyl Compounds. SOV/62-59-9-17/40  
Communication 5. Polymerization of Vinyl Succinimide

parent, and becomes porous and opaque when larger quantities of BP are used. The polymerization of the solving agents (dichloroethane, benzene, methyl alcohol, and water) rapidly occurred at 85° even in diluted solving agents and the yield was good. (Table 3). As particular properties of the obtained polymers the following 2 have been established: limited solubility in organic solving agents and a low stability in water (Table 3). There are 3 figures, 4 tables, and 5 references, 3 of which are Soviet.

ASSOCIATION: Leningradskiy tekhnologicheskii institut im. Lensoveta (Leningrad  
Institute of Technology imeni Lensovet)

SUBMITTED: January 8, 1958

Card 2/2

5 (3)  
AUTHORS:

Nikolayev, A. F., Ushakov, S. N.,  
Grinburg, R. B.

SOV/62-59-9-18/40

TITLE:

Polymerization and Copolymerization of N-Vinyl Compounds.  
Communication 6. Simultaneous Polymerization of Vinyl Succinimide  
and Methyl Methacrylate

PERIODICAL:

Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk,  
1959, Nr 9, pp 1631 - 1635 (USSR)

ABSTRACT:

The appropriate publications have not yet discussed the copoly-  
merization of vinyl succinimide with methyl methacrylate. The  
present paper describes this copolymerization and lists several  
properties of the copolymer. To establish the conditions of the  
copolymerization, the influence of the temperature (50, 65°,  
Fig 1), and the influence of the composition of the initial com-  
ponents on the rate of the copolymerization reaction was in-  
vestigated (the experiment lasted 1, 2, and 3 hours, Fig 2). The  
investigations established that methyl methacrylate is the more  
active component in the copolymerization. The analysis data, ta-  
ble 1, show that a small part of the succinimide was consumed at  
the copolymerization. For the acceleration of the reaction the  
initiator benzoyl peroxide (BP) and azodi-isobutyronitrile (AN)

Card 1/3

Polymerization and Copolymerization of N-Vinyl Compounds. SOV/62-59-9-18/40  
Communication 6. Simultaneous Polymerization of Vinyl  
Succinimide and Methyl Methacrylate

were additionally applied. The data obtained (Fig 3) show that AN initiates more efficiently at low temperatures, this difference is equalized by the increase of the reaction temperatures. The authors investigated the concentration relations of the basic substances 5:1, 2:1, 1:1, 1:2. Applying the initiator BP in the ratio 1:1 of the basic component and at 65-70° a yield of 95-98% was obtained within 7-8 hours. The copolymer was a thin, porous film after the evaporation of the solvent. This film rapidly softens when warmed. The authors also investigated the molecular weight, water-repelling capacity, temperature stability, solidity, density, and tensile strength of the products obtained, and it showed that with an increase of the vinyl succinimide content the three first-mentioned values decrease, while the latter increase. The copolymer with 50% of vinyl succinimide content has a greater temperature stability at 30° than pure methyl methacrylate. There are 3 figures, 3 tables, and 4 references, 3 of which are Soviet.

Card 2/3

Polymerization and Copolymerization of N-Vinyl Compounds. SOV/62-59-2-18/40  
Communication 6. Simultaneous Polymerization of Vinyl  
Succinimide and Methyl Methacrylate

ASSOCIATION: Leningradskiy tekhnologicheskii institut im. Lensovet (Leningrad  
Institute of Technology imeni Lensovet)

SUBMITTED: January 8, 1958

Card 3/3

SOV/86-32-3-36/43

5(3)

AUTHORS: Ushakov, S.M., Nikolayev, A.F., Torontseva, A.M., Trizna, M.S.

TITLE: The Synthesis of Monoalkylmaleates (Sinter monoalkilmaleinatsy)

PERIODICAL: Zhurnal prikladnoy khimii, 1959, Vol XXXII, Nr 2, pp 667-672 (USSR)

ABSTRACT: The derivatives of dibasic acids polymerize with various mono- and divinyl compounds. The monoesters of maleic acid are investigated here. They are prepared by the reaction of maleic anhydride and primary, secondary, tertiary alcohols of the aliphatic, cyclic and aromatic series. Monoethyl maleate is obtained from maleic anhydride and absolute ethyl alcohol. It is separated from the reaction mixture by potash, ether, alcohol, diluted hydrochloric acid etc. The optimum temperature for the reaction is 80°C. A lowering of the temperature to 60°C reduces the reaction rate considerably. A temperature increase leads to decomposition of the monoester. The monoesters of the maleic acid are colorless, transparent, viscous liquids with a characteristic odor. They are soluble in

Card 1/2

SCV/80-32-3-36/45

The Synthesis of Monoalkylmaleates

storing but not to heating. Their specific weight decreases with the increase of the molecular weight of the alcohol (Table 3).

There are 3 tables and 7 references, 1 of which is Soviet, 3 English, 2 American and 1 Swiss.

SUBMITTED: Janua 7, 1958

Card 2/2

SOV/20-128-1-31/53

5(3)

AUTHOR:

Ushakov, S. N. Corresponding Member AS USSR

TITLE:

Some Reactions in the Chains of Vinyl Alcohol Copolymers

PERIODICAL:

Doklady Akademii nauk SSSR, 1959, Vol 128, Nr 1, pp 117-120 (USSR)

ABSTRACT:

The author in collaboration with his co-workers I. A. Arbuzova, S. A. Plotkina and I. Santo (Refs 1 and 2) suggested a series of new cross-linking divinyl agents, which yield good results in the heteropolymerization with acryl derivatives and vinyl esters. Diallyl acetates of various aldehydes (formal, ethylal, butylal) were thoroughly investigated in their function as active agents. Diethers of the methylol croton amide and methylene-bis-croton amide belong to another group of particularly active agents (Author and Ye. M. Lavrent'yeva, K. S. Podgorskaya, Ref 3). A far more complete process of the formation of polymers is warranted by cross-linking of long, linear macromolecules by condensation of the reagent functional groups contained in the chain. In order to avoid ring formations in the copolymeric chain it is expedient to introduce the functional groups by heteropolymerization. As an interesting example for such processes serves the copolymerization suggested

Card 1/3



SOV/20-128-1-31/58

## Some Reactions in the Chains of Vinyl Alcohol Copolymers

and carried out by the author together with Ye. M. Lavrent'yeva and K. S. Podgorskaya of vinyl acetate and other vinyl esters with methylol croton amide and croton amide. Copolymerization with methylol croton amide may be carried out in the solid state or in liquid in the presence of initiators of the radical polymerization such as benzoyl peroxide, acetyl benzoyl peroxide, and dinitro azoisobutyric acid. The linear heteropolymers thus obtained are thermoactive and on heating form non-melting and insoluble polymers of steric structure. On the basis of an investigation of the mechanism of the radical polymerization of vinyl acetate with croton amide and methylol croton amide carried out by the author in co-operation with B. L. Trukhmanova the copolymerization constants of these systems could be determined. The new copolymers of vinyl esters and vinyl alcohol with methylol croton amide (and croton amide) may find a wide range of application from the practical point of view. Thermically treated copolymers are of a far higher mechanical stability than the pure polyvinyl alcohol. They are absolutely insoluble both in the cold and boiling water. The thermoactivity characteristic of the linear copolymer and caused by the introduction of links with non-adjacent functional groups offers

Card 2/3

SOV/20-128-1-31/58

Some Reactions in the Chains of Vinyl Alcohol Copolymers

great prospects for their technical applicability. The new group of heteropolymers with crotonic acid derivatives is of the greatest interest for the manufacture of covers, synthetic materials and synthetic fibers. There are 1 table and 6 Soviet references.

ASSOCIATION: Institut vysokomolekulyarnykh soyedineniy Akademii nauk SSSR  
(Institute of High-molecular Compounds of the Academy of Sciences, USSR)

SUBMITTED: May 18, 1959

Card 3/3

AUTHOR: Ushakov, S. N., Corresponding Member  
of the Academy of Sciences USSR

S/030/60/000/03/008/044  
B015/B008

TITLE: New Soviet Synthetic Fiber "Vinilon" ✓

PERIODICAL: Vestnik Akademii nauk SSSR, 1960, Nr 3, pp 52-54 (USSR)


TEXT: In the paper under review the author reports on the development of the manufacture of this fiber in the USSR, for which acetylene is used as the main raw material. The essential values of the properties of the fiber from polyvinyl alcohol, called "Vinilon", are mentioned in the table. The Institut vysokomolekulyarnykh soyedineniy Akademii nauk SSSR (Institute of High-molecular Compounds of the Academy of Sciences USSR) worked out the production methods for polyvinyl acetate and polyvinyl alcohol. Experimental batches of polyvinyl alcohol were produced in the experimental plant of the Nauchno-issledovatel'skiy institut polimerizatsionnykh plastmass (Scientific Research Institute for Synthetic Polymerization Materials). Studies concerning the production of new types of fibers on the basis of polyvinyl alcohol are carried out at these 2 institutes, the papers by S. N. Ushakov and Ye. M. Lavrent'yeva who first used thermoreactive copolymers of vinyl alcohol especially with methyl croton amide and croton amide, being mentioned. Finally the author expresses the hope that

Card 1/2

New Soviet Synthetic Fiber "Vinilon"

S/030/60/000/03/008/044  
B015/B008

the joint research of the Institute of High-Molecular Compounds, the competent special research institute and an industrial enterprise will make it possible to produce new synthetic fibers on the basis of copolymers of vinyl alcohol. There is 1 table.



Card 2/2

USHAKOV, Sergey Nikolayevich; MATVEYEV, I.I., kand.khim.nauk, otv.red.  
[deceased]; CHIZHOV, A.A., red.izd-va; KHUGLIKOVA, N.A.,  
tekhn.red.

[Polyvinyl alcohol and its derivatives] Polivinilovyi spirt  
i ego proizvodnye. Moskva, Izd-vo Akad.nauk SSSR. Vol.2.  
1960. 866 p. (MIRA 14:1)  
(Vinyl alcohol)

USHAKOV, S.N., prof. (Leningrad); SZANTO, Istvan (Budapest)

Investigations on crosslinked polymers. I. Preparation of crosslinked polyvinyl acetate and polyvinyl alcohol, and characterization of their properties. Acta chimica Hung 24 no.3:343-356 '60. (EEAI 10:3)

1. Technological Institute, L.T.I., Leningrad (for Ushakov). 2. Central Research Institute for Chemistry, Hungarian Academy of Sciences, Budapest (for Szanto)

(Polymers and polymerization)	(Vinyl alcohol)
(Vinyl acetate)	(Formaldehyde)
(Butyraldehyde)	(Aldehydes)
	(Acetals)
	(Acetates)

S/020/60/134/003/032/033/XX  
B016/B060

AUTHOR: Ushakov, S. N., Corresponding Member AS USSR

TITLE: Production of Films, Threads, Poroplasts, and Thixotropic Gels From Iodine Complexes of Polyvinyl Alcohol and its Copolymers

PERIODICAL: Doklady Akademii nauk SSSR, 1960, Vol. 134, No. 3,  
pp. 643 - 646

TEXT: The author first describes the formation of "deposits" from iodopolyvinyl alcohol, which, while being gradually resorbed, give rise to local zones of antimicrobial effect in the organism, and retain this effect only provided thixotropic gels are used for the purpose. The application of films, threads, poroplasts, and thixotropic gels from polyvinyl alcohol had not been described before. The author established that iodopolyvinyl alcohol is thermally unstable, decomposes at 40-60°C, and loses its antimicrobial properties. The usual methods of preparing the said products of iodopolyvinyl alcohol are therefore inexpedient and not applicable for any practical purpose. The author found a possibility

Card 1/4

Production of Films, Threads, Poroplasts, and Thixotropic Gels From Iodine Complexes of Polyvinyl Alcohol and Its Copolymers

S/020/60/134/003/C32/C33/XX  
B016/B060

of getting around all these difficulties by having the iodine complex of polyvinyl alcohol result from polyvinyl alcohol through heterogeneous reaction on the surface of films, threads, and poroplasts. For this purpose they are immersed into an iodine solution. Polyvinyl alcohol selectively sorbs iodine from iodine solutions in aqueous solutions of iodine salts (potassium, ammonium, and other iodides). This gives rise to colored complexes. The author describes this reaction and its most favorable conditions, and specifies the sizes of films and threads used. The method described here is also suited for the production of pulverulent iodopolyvinyl alcohol. It has certain advantages over the usual methods (Ref.4). The author's method acquires a special significance when using fine-disperse powders of "cross-linked", insoluble polyvinyl alcohol, which are, among other things, used for disinfecting wounds or sterilizing water. The author has worked out such powders as are specially suited for treatment with the heterogeneous reaction. He used the method of emulsion copolymerization of vinyl acetate with 0.1-3.0 mole% of tetrareactive compounds which, together with vinyl acetate, form

Card 2/4



Production of Films, Threads, Foams, Lasts, S/020/60/134/003/032/033/XX  
and Thixotropic Gels From Iodine Complexes B016/B060  
of Polyvinyl Alcohol and Its Copolymers

heteropolymers. As tetraactive compounds, the author suggested diallyl acetals (jointly with I. Arbuzova and S. Plotkina, Ref.6) or derivatives of crotonic acid (methylol crotonamide diether, methylene-bis-crotonamide). The resulting emulsion was destroyed by the addition of NaCl electrolyte; the powder was filtered off, dried in vacuum, and subjected to a heterogeneous methanolysis with absolute methanol. The complete heterogeneous saponification did not destroy the acetal bonds of diallyl formal bridges between the chains, and the spatial "cross-linked" copolymer structure remained unaltered. Emulsion polymerization of vinyl acetate with other diallyl acetals proceeds in an analogous manner. These high-disperse powders (particle size up to micron fractions) cannot be produced by the comminution of polymers, and are specially suited for the production of iodine complexes. V. Mokhnach, S. Andreyev, M. Litvinov, L. Borisov, Ye. Lavrent'yeva, K. Podgorskaya, and I. Santo are mentioned. There are 9 references: 5 Soviet, 1 Canadian, 1 British, and 3 German.

Card 3/4

Production of Films, Threads, Poroplasts,  
and Thixotropic Gels From Iodine Complexes  
of Polyvinyl Alcohol and Its Copolymers

S/020/60/134/003/C32/C33/XX  
B016/B060

ASSOCIATION: Institut vysokomolekulyarnykh soyedineniy Akademii  
nauk SSSR (Institute of High-molecular Compounds of the  
Academy of Sciences USSR)

SUBMITTED: April 21, 1960

Card 4/4

84830

S/020/60/134/005/018/023  
B016/B054

15.8116 2209 only

11.2217  
AUTHORS:

Ushakov, S. N., Corresponding Member AS USSR and  
Belogorodskaya, K. V.

TITLE:

On the Synthesis of Silicon Derivatives of Polyvinyl  
Alcohol

PERIODICAL:

Doklady Akademii nauk SSSR, 1960, Vol. 134, No. 5,  
pp. 1115-1118

TEXT: As there are no data published on the production of various  
silicon derivatives of polyvinyl alcohol which are used to modify its  
properties, the authors tried to produce these derivatives (general  
formula  $-\text{CH}_2-\underset{\text{OSiR}_3}{\text{CH}}-\text{CH}_2-\underset{\text{OH}}{\text{CH}}-\text{CH}_2-$ , where R is an alkyl-aryl or aralkyl).

For this purpose, they used the following reactions: a) of chloro silanes  
with polyvinyl alcohol as well as with its alcoholates in a heterogeneous  
medium; b) of chloro silanes with partially saponified polyvinyl acetate  
in a homogeneous medium; c) of trialkyl aminosilanes with polyvinyl

Card 1/3

84830

On the Synthesis of Silicon Derivatives of  
Polyvinyl Alcohol

S/020/60/134/005/018/02;  
B016/B054

alcohol in a pyridine medium. In the case a), there are difficulties due to the good reactivity of chloro silanes with water, pyridine, formamide, and other solvents of polyvinyl alcohol. In the heterogeneous reaction under a), the finely ground powders of polyvinyl alcohol, its alcoholate, and its alkaline derivative were suspended in benzene, mixed with trimethyl chlorosilane, and stirred at 20-70°C for 7-24 h. This did not lead to a noticeable substitution of the hydroxyl groups of the alcohol by alkyl silicon radicals. Further, partially saponified polyvinyl acetates (case b)) were used which maintain their solubility in benzene. To attain the latter reaction, the alcoholysis must be carried on to a maximum content of 10 mole% of hydroxyl groups in the polyvinyl acetate chain. The reaction under b) was carried out in benzene or in a benzene-dioxane mixture. The medium was absolutely anhydrous. The resulting HCl was bound with suspended  $MgCO_3$ , which is of great importance. The product obtained was precipitated from a filtered solution with petroleum ether, purified by dissolving it twice in dioxane, and precipitated with water (Table 1). Thus, 50-70% of all free hydroxyl groups of the partially saponified polyvinyl acetate were substituted. No noticeable destruction occurs. The resulting copolymers with a Si content of 4.8% have an

4

Card 2/3

00000

On the Synthesis of Silicon Derivatives of  
Polyvinyl Alcohol

S/020/60/134/005/018/023  
BO:6/BO54

increased vitrification temperature:  $T_{\text{vitr}} = 38^{\circ}\text{C}$ . In the case c), the same apparatus was used as in the case b) (a three-neck flask with re cooler). The pyridine used was absolutely dry, and protected from air moisture. Previously, polyvinyl alcohol was swelled in pyridine for 18-20 h. The reaction mass is completely homogenized within 1.5-2 h. The reaction product was precipitated with various organic liquids since its solubility strongly fluctuates depending on the degree of substitution. The authors found that under the above conditions an organosilicon ether of polyvinyl alcohol is formed. Table 2 shows results of some special experiments of the reaction of the above ether with triethyl aminosilane. Hence, it appears that triethyl silyl ethers of polyvinyl alcohol were obtained with different degrees of substitution. Table 3 shows the solubility of some products obtained, Table 4 lists their properties. There are 4 tables and 4 references: 3 Soviet and 1 US.

SUBMITTED: June 10, 1960

Card 3/3

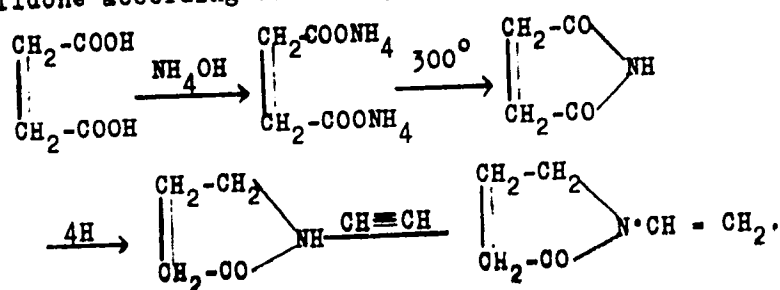
S/062/61/000/005/007/009  
B118/B220

AUTHORS: Ushakov, S. N., Davidenkova, V. V., and Lushchik, V. V.

TITLE: Synthesis of vinyl pyrrolidone

PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk, no. 5, 1961, 901 - 905

TEXT: Starting from succinic acid, the authors synthesized in 1952 vinyl pyrrolidone according to the equation



Card 1/4

S/062/61/000/005/007/009  
B118/B220

## Synthesis of vinyl...

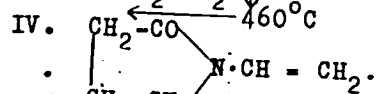
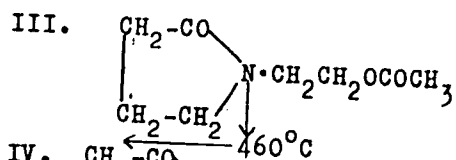
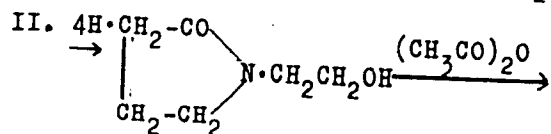
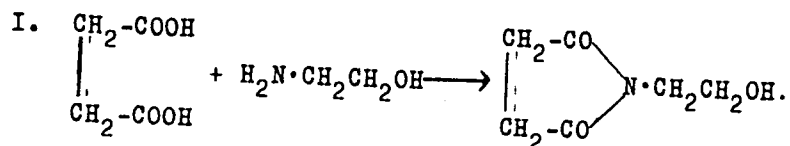
The succinimide was obtained from the ammonium salt of the succinic acid at 300°C and, after purification, reduced electrolytically to pyrrolidone on lead electrodes (80 to 90 mA/cm) in 50 % sulfuric acid. Vinylizing of pyrrolidone was effected in dioxane solution in the presence of potassium pyrrolidone in the autoclave under a pressure of 15 to 25 atm and at 125 to 132°C. This method is easier than that proposed by W. Reppe (Ref. 1, Polyvinylpyrrolidon, 1954, Berlin). The vinyl pyrrolidone produced was used for the synthesis of polymers which in the Leningradskiy institut perelivaniya krovi (Leningrad Institute for Blood Transfusion) have proved to be good blood substitutes. Independently of this paper and almost at the same time, data were published concerning the synthesis of pyrrolidone from succinic acid and ammonia via succinimide (G. 1953, 9185; Rev. Plastic, 2, 110, 132). But also for this modified synthesis, the last part of the vinylizing, effected under pressure and using acetylene, is rather difficult. In a series of cases it was, therefore, of advantage to realize the synthesis without acetylene and without increasing the pressure (e. g. according to the equation by B. Puetzer et al., J. Amer. Chem. Soc. 74, 4956 (1952)). Unlike the USA Patent 2669570, the authors of the present paper succeeded in synthesizing vinyl pyrrolidone from succinic acid by

Card 2/4

Synthesis of vinyl...

S/062/61/000/005/007/009  
B118/B220

using neither pressure nor acetylene:



Card 3/4



Synthesis of vinyl...

S/062/61/000/005/007/009  
B118/B220

Based on succinic acid and using easily obtainable reagents (monoethanol amine and acetic anhydride), they contained, thus, vinyl pyrrolidone by pyrolysis in 4 stages, without acetylene and increased pressure (yield: 52 % of the theoretical-one). There are 9 references: 5 Soviet-bloc and 4 non-Soviet-bloc.

ASSOCIATION: Institut vysokomolekulyarnykh soyedineniy Akademii nauk SSSR  
(Institute of High Molecular Compounds of the Academy of  
Sciences USSR)

SUBMITTED: April 9, 1960

Card 4/4

25217

S/062/61/000/007/007/009  
B117/3215

15-8050

AUTHORS: Nikolayev, A. F., Ushakov, S. N., and Daniel', N. V.  
TITLE: Polymerization and copolymerization of N-vinyl compounds  
PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk, no. 7, 1961, 1330-1336

TEXT: Information 8. Polymerization of vinyl succinimide in aqueous solution. This paper continues the study of polymerization of vinyl succinimide in aqueous solution in the presence of an initiator soluble in water. Potassium persulfate was used. Vinyl succinimide was prepared and purified by the method of Ref. 9 (S. N. Ushakov i A. F. Nikolayev, Izv. AN SSSR. Otd. khim. n. 1956, 226). Potassium persulfate was analyzed according to a method suggested for determining peroxide compounds (Ref. 10: A. Schwicker, Z. analyt. Chem. 74, 433 (1928)). For the polymerization of vinyl succinimide a flask with a mixer and mercury sealing, reflux condenser, and thermometer were used. A number of experiments were conducted in nitrogen atmosphere or without mixing. By adding formaldehyde (in the form of formalin) and uric acid, the length of

Card 1/5

Polymerization and copolymerization<sup>25217</sup>...

S/062/61/000/007/007/009  
B117/B215

the polymer chains was regulated, and their cross-linking eliminated. The reaction temperature was maintained at 70° and 80°C with an accuracy of  $\pm 0.2^\circ$ . A 10% aqueous vinyl succinimide solution was used in all experiments. The reaction was continued until a 95-98% transformation was attained. Examination of the polymerization under static conditions and with stirring showed that the rate of the process largely depends on hydrodynamic conditions. Vigorous mixing completely inhibits polymerization. Slight mixing slows the process down. At an initiator concentration of more than 0.2% and a temperature of 70-80°C, the reaction proceeds fast only without mixing. Experiments in nitrogen atmosphere showed that polymerization in this case was normal with stirring and also under static conditions. Hence, it can be seen that atmospheric oxygen has an inhibitory effect on the polymerization of vinyl succinimide under the above reaction conditions, especially at temperatures below 70°C and with stirring. It was expected that oxygen loses its inhibitory effect when the reaction temperature is elevated. Actually, polymerization of vinyl succinimide is fast at 80°-90°C and at any mixing rate. At lower temperatures, however, no polymers were formed. By adding 16% or more formalin and 10% or more uric acid, a polymer forms which is soluble in

Card 2/5

Polymerization and copolymerization<sup>25217</sup>

S/062/61/000/007/007/009  
B117/3215

chlorinated hydrocarbons, especially methylene chloride and chloroform (Table). The necessity of using chain propagators in the polymerization of vinyl succinimide indicates that the polymeric vinyl succinimide radical is most reactive. From this results its ability of propagating the chain via the polymer. By elevating the temperature from 65° to 80°C the polymers become better soluble. This is due to the reduced molecular weight of the resulting polymer. The polymerization of vinyl succinimide in aqueous solution is fast and complete in the presence of potassium persulfate. An analysis of the dependences of polymerization leads to the following conclusions: (1) In water, the water-soluble initiator decomposes into primary radicals, part of which is recombined. The greater part, however, is bound by vinyl succinimide. (2) Polymerization requires a strongly effective initiator. (3) During the reaction polyvinyl succinimide is separated from the solution. This process, however, does not affect the increase in viscosity of the reaction medium. (4) The full rate of polymerization is proportional to the square root of the initiator concentration not only in the initial stage, but also at high-degree transition. This conclusion is confirmed by experimental data in the range of the potassium persulfate concentrations examined, namely,

Card 3/5